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Report No. IITRI C6093-13
(Final Report)

DETECTION AND IDENTIFICATION OF
CHEMICAL SIGNATURES

Ballistic Research Laboratories
Aberdeen Proving Ground, Md.

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IIT RESEARCH INSTITUTE

Report No. IITRI C6093-13
(Final Report)

DETECTION AND IDENTIFICATION OF CHEMICAL SIGNATURES

January 8, 1966 through April 7, 1969
Contract No. DA-18-001-AMC-954(X)

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FOREWORD

This report discusses the experimental procedures used and results obtained during work under Contract DA-18-001-AMC-954(X) entitled, "The Detection and Identification of Chemical Signatures".

Personnel who contributed to this program include, B.K. Krotoszynski, A. Dravnieks, Wanda Bahmet, H.W. Neumann, N.S. Snaw, T.A. Stanley, and Joyce Burton.

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DETECTION AND IDENTIFICATION OF CHEMICAL SIGNATURES

ABSTRACT

In the work on the detection and identification of human chemical signatures experimental and computerized statistical techniques were first developed, tested, and standardized. The experimental techniques solved the problems of sample collection, recovery, injection into the gas chromatograph, calibration and characterization of signature components. The sample collections were based on the principle of equilibration and were performed by means of Apiezon L-coated Teflon powder in the form of a fluidized bed. The collected samples were transferred into a special injector needle by means of an IITRI-designed transfer system. The injection of samples into the gas chromatograph was performed in a timed, automatic and reproducible manner with a specially constructed injection system. The calibration method permitted correlation of the concentrations of known signature components in air with their respective peak areas. The sensitivity of this novel process is of the order of one part per billion (by volume) of an organic component in air.

The developed techniques were used to collect and analyze vapor samples from humans temporarily isolated from the environment by being placed in a special glass cell containing high purity air. The glass cell was fitted with low-adsorptive Teflon gaskets and a Teflon coated resting platform. The gas chromatographic blanks of air obtained from the system contained a few small size peaks in the high-volatility range of the chromatograph when analyzed by the developed process.

In 65 human chemical signatures collected and analyzed in the packed 10 ft Carbowax 20M column at 125°C, 32 to 48 peaks per signature were observed. Statistical pattern analysis using stepwise discriminant analysis on these human chemical signatures which included blocks of Caucasian males, Indian (from India) males, Caucasian males, and on 10 chemical signatures of assorted

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environments showed that valid discriminant functions can be obtained utilizing 5 to 15 of the gas chromatographic ranges. These findings indicated that source-type-characteristic features exist in the moderately resolved composition of vapors from humans obtained gas chromatographically. These features can be used as a basis for detection and identification processes.

Higher resolution gas chromatography at several temperature levels in a 200-ft support-coated open tubular Carbowax 20M column exhibited typically 100 to 130 peaks. Twenty-five of these, located in the gas chromatographic ranges where peaks were most frequently observed in the lower-resolution human chemical signatures, were re-analyzed in a specially designed two-column gas chromatograph in a 50-ft Apiezon L column. Each one of the 25 peaks consisted of 3 to 10 chemical components, or more than 150 components (total) when examined in the two column apparatus. The polarity of the components indicated the presence of considerable amounts of hydrocarbons, carbonyl compounds, and others, such as alcohols. Other materials, exceeding the alcohols in their gas chromatographic polarity, are probably bifunctional compounds or those with a functional group and more than one double bond in the molecule. Analysis of collected samples in a specially designed three-column gas chromatograph utilizing computerized data interpretation similarly indicated a great complexity of composition.

Mass spectrographic identification of the components, with a few exceptions (moth-proofing compounds were found) was not successful. The analysis with the two-column gas chromatograph demonstrated that the principal reason for this difficulty was the lack of resolution, even by analysis using a single 200-ft tubular column. Satisfactory future identification should be possible using the resolved output from the combination 200-ft Carbowax 20M/50-ft Apiezon L columns in series.

The raw data were tabulated in terms of Kovats retention index and are listed in the Appendixes.

1.0 INTRODUCTION

In the previous project, "Detection and Identification of Chemical Signatures," Contract DA-18-001-AMC-954 (X), the following work was conducted: (1) a glass cell apparatus was designed and constructed for temporary isolation of human subjects in a stream of highly purified air. (2) a vapor collection, sample transfer, and gas chromatographic procedures were developed for obtaining a gas chromatographic representation of organic components in the effluents from the temporary isolated subjects, and (3) several representative gas chromatograms of several subjects of different races and both sexes were obtained.

The objective of the present project was (1) to further improve the vapor collection and evaluation techniques, (2) obtain a sufficient number of chemical signatures (gas chromatographic representations) of vapors from a variety of subjects so that some generalizations would become possible, (3) to characterize, as much as possible, those human vaporous effluent components that may be most suitable for identification of human vapors in various environments, and (4) to prognosticate on the most promising approaches to the effluent composition characterization processes that might be suitable for identification of human-related vaporous components.

As the work progressed, the variability of the chemical signatures among the individuals and the complexity of the vapor composition emerged as the dominant features complicating the tasks (3) and (4) of the objectives. Consequently, much effort had to be spent on techniques of data obtaining and interpretation, to facilitate the tasks (3) and (4). The work consisted of considerable improvement in the techniques in statistical pattern analysis to direct attention to those gas chromatographic features which are most human-related and high-resolution gas chromatography and some mass spectrography to better characterize the chemical nature of the organic fractions of the effluents from humans.

2.0 SELECTION OF EXPERIMENTAL APPROACHES

The previous work indicated that human chemical signatures vary widely among the individuals and to a considerable extent in the same individual and contain many organic components. The volatility of the components encountered covered a broad range, beginning with volatile substances apparently belonging to the lowest members of the homologous series and expected to occur in most atmospheres and extending to substances with much lower volatility such as expected from organic substances with ten or more carbon atoms in molecule. It became obvious that the human-character components may easily occur in the heavier (lower volatility) sections of gas chromatographic spectrum and that the chromatographic peaks observed most likely consist of more than one substance. At that juncture, two courses were possible: (1) work toward improvement of gas chromatographic resolution with detailed study of components encountered in a few individuals, or (2) survey of chemical signatures of many individuals and some environments with intention to locate those gas chromatographic features which would be most suitable for discrimination of humans vs environments so that the later higher-resolution studies could be concentrated on the significantly discriminating ranges.

The second of these routes was selected, to avoid the possibility of wasting initial efforts on identification of those components which later might be found not to be emitted by many humans or be not particularly suitable for detection of vapors from humans in environments. Since the chemical signatures extended into the range of heavier substances, the air sampling technique had to be modified before data accumulation could begin to permit sufficient collection of substances with lower vapor pressures.

The above considerations guided the experimental approach through the following stages:

1. Fluidized bed collector method with suitable vapor transfer devices and calibration apparatus were developed to permit systematic and reproducible chemical signature collection and transfer to the gas chromatograph.
2. After preliminary experimentation with the fluidized bed collectors for human chemical signature collection, standardized samples of organic vapors were obtained in 65 samplings from individuals of both sexes and several races (with some individuals repeated) and in 10 samplings from several environments.
3. The collected samples were analyzed using 10-ft packed bed Carbowax 20M column at 125°C, obtaining 75 gas chromatographic patterns.
4. The patterns were submitted to computerized statistical analysis, to detect those features that are most suitable for discrimination of different vapor source classes e.g., humans vs environment, different groups of humans (males vs females, Indian males vs Caucasian males, etc.). Several statistical approaches and rationales were used, and resulted in pointing out those gas chromatographic ranges which contained components potentially useful for source class discrimination.
5. Several methods of high-resolution gas chromatography were developed to increase the component resolution to a degree where mass spectrographic analysis of individual components would be possible. These methods progressed through: 50-ft and 200-ft support-coated open tubular single-column gas chromatograph; a two-column chromatograph where peaks from the first high-resolution polar column could be individually injected into a second, non-polar column; and a three-column gas chromatograph where the entire pattern could be obtained in polar and non-polar columns and correlated using specifically written computerized peak-matching program. Because of the high complexity of the sample, the three-column patterns lost considerable detail and appeared to present principally the gross composition of the chemical signature. The two-column device was judged to yield most information, and the chemical signature of one individual was studied in detail by repeated analyses with this device, inspecting the ranges indicated significant by the statistical analysis.
6. The initial attempts to mass spectrographically identify the components after resolution in a 50-ft support-coated open tubular Carbowax 20M column did not yield, with one exception, clearly identifiable components. Later work with similar single 200-ft Carbowax 20M column and with the two-column device indicated that

even the peaks emerging from the 200-ft column were further separable into 2 to as many as 10 components. This explained the difficulty experienced in the earlier mass spectrographic attempts of the component identification and pointed the way, in the form of the two-column device, for obtaining gas chromatographic resolution sufficient for more successful mass spectrographic identification.

7. An initial listing system for classification of the components of the chemical signatures in terms of retention (Kovats) indexes was adapted, and some considerations for chemical signature detector devices were evolved.

3.0 EXPERIMENTAL

The various subsections of this chapter deal with the chemical signature collection and vapor transfer techniques, calibration, subject selection, statistical pattern analysis experiments, and several techniques for high-resolution analysis of the collected samples.

3.1 Glass Cell Apparatus

The 18-in I.D. cylindrical glass cell, with Teflon ring seals, Teflon-coated end plate, and a Teflon-coated steel frame stretcher for the support of human subjects in the supine position in the middle of the cell has been described in detail in the previous report.*

For the chemical signature collection, the subjects were enclosed in the cell for 45-min in a stream of highly purified air. The air was taken from hospital grade cylinders. Experience indicated that the commercially available hospital grade air contained organic impurities in varying concentrations sufficient to make the analysis of the vapors emitted by humans uncertain and cumbersome.

Experiments were conducted to design purification procedure that would reduce the contaminant level in the air fed to the cell to a practical minimum. Air first passes through a particle filter (Vetrical, Type VF-6, 47mm Dia, Pore size .45U. Gelman Instrument Company, Ann Arbor, Michigan) then in downflow through an activated charcoal bed (Figure 1) cooled in its lowest one third section by a dry-ice-propanol mixture. The gradual cooling of air in the charcoal bed prevents aerosol formation, since vapors adsorb at approach to their dew point and before an overcooling that can lead to aerosol formation occurs. The diameter of the carbon bed was 4-in and the rate of air flow 0.75 l/sec. The

*Report IITRI No. U6012-4, Contract DA-11-022-AMC-1775(X)

charcoal bed adsorber was activated and stored at 200°C in nitrogen stream between uses, so that each chemical signature collection experiment began using a freshly reactivated adsorber.

The purified air entered the glass cell at very low humidity level, but sufficient humidity was picked up from human subject to result in 40-50 percent relative humidity at the exit of the cell. The exit air temperature typically was 23° to 25°C. Higher humidities for which provisions were made in the initial design of the air preparation train were avoided, since the subjects tended to begin perspiring profusely, and the excess humidity resulted in clouding the glass and in difficulties with water condensation in the vapor collectors kept at room temperature.

The content of oxygen in the air decreased by less than 2 percent absolute in passage through the cell when it was occupied by a subject.

3.2 Fluidized Bed Collectors

The organic vapor collection was conducted using the principle of absorption in highly purified grease films. This principle, used in gas chromatography in the form of "pre-column" collectors, permits collection of organic materials from vapor phase without interfering accumulation of water vapor, if non-polar greases are used. However, in its usual form of a packed bed, consisting of grease or oil-coated inert powders, it has certain disadvantages: the vapors of substances that are more volatile and less soluble in the grease can saturate the collector upon passage of certain limited air volume and the excess begins to pass through the collector, while the less volatile or more oil-soluble vapor components may still continue to be fully removed by the collector from the air passing through the collector. Therefore, at any given volume of air passed, some vapor components will reach the equilibration concentration and their amount in the collector will not further increase, regardless how much more air is passed. Other components will continue to accumulate until their specific

equilibration conditions will be met.

The maximum amount of a substance that can be collected from its vapors in air is determined by several physicochemical variables combined as follows:

The number of molecules of certain species collected is

$$n = 6 \cdot 10^{23} \frac{d}{M} \cdot \frac{1}{\gamma} \cdot \frac{n_a}{n_0} \cdot V \quad (I)$$

Here d and M are the density and the molecular weight of the film substance, respectively; γ is the activity coefficient of the species in the film; n_a is the concentration, molecules/cm³, of the species in the film; n_0 is the concentration in gas at saturation at the same temperature; and V is the volume, cm³, of the film phase in the sampler.

The activity coefficient is large for cases when the vapor is polar while the collecting phase is non-polar. For the same vapor and collecting phase, the amount collected is proportional to the concentration of this vapor in air (n_a) if the collector temperature and consequently n_0 are maintained constant.

For convenience, it can be pointed out that n/n_a represents the volume of air, in cm³, from which the particular vapor can be fully extracted, and by rearranging Eq. 1,

$$\frac{n}{n_a} = 6 \cdot 10^{23} \frac{d}{M} \cdot \frac{1}{\gamma} \cdot \frac{1}{n_0} \cdot V \quad (II)$$

it is seen to be essentially independent of the concentration of the vapor in air, since all terms on the right side are either concentration-independent, or occasionally only partially dependent as is the case with the activity coefficient. The ratio n/n_a is equivalent to the gas chromatographic retention volume of the specific component in the collecting phase at collection temperature--usually room temperature.

In the later gas chromatographic analysis, unless it is conducted using the same stationary phase as in the collector

(Apiezon L), it is impossible to determine for which components the air volume passed through the collector has exceeded the n/n_g volume. The most convenient solution for this indeterminacy was found in developing a fluidized bed collector. Here the collecting phase, in the form of Apiezon L films dispersed on Teflon particles, is placed in a capsule through which air to be sampled is flown at a high rate. The vapors in air reach each particle easily, since the powder is in a violent motion, and an equilibration is reached on all particles essentially simultaneously. For fluidized bed samplers coated with 10-wt percent of Apiezon L, equilibration was reached within 20-30-min. To assure equilibration, the collection was continued for 45-min.

The fluidized bed material is contained in a cylindrical glass column, 5-in long and 1-1/4-in wide (Figure 2). The column is provided with stainless steel screen discs at each of its ends. The diameter of the screen openings is selected to retain the powder in the column during vapor collection and to exert minimum resistance to the airflow.

The fluidized bed material is introduced into the column through a side arm, which is then sealed off. The bed material remains in the column during its conditioning, vapor collection, and vapor transfer. The bed material consists of 10 g of Fluoropak 80 coated with 10% Apiezon L grease; therefore a typical fluidized bed sampler contains 1 g of Apiezon L in the form of a film a few microns thick. From many conventional gas chromatographic phases tried, only Apiezon L permitted later recovery of dissolved organics without artifacts (extra peaks) from decomposition of the trapping phase itself.

Before each vapor collection, the bed material is conditioned by heating the bed at 80°C and flushing it with pure helium gas until all volatile impurities are removed. The conditioning efficiency is established by collecting residual impurities in an injector needle (cf Section 3.3) and gas chromatographing the collected sample. The bed material is considered clean when the

residual impurities produce peaks of negligible areas. Temperatures higher than 80°C maintained for 1 hr led to incipient thermal decomposition of Apiezon L with formation of volatile compounds.

The bed material is then exposed to air carrying the vapors to be collected and analyzed. Air is either pushed or pulled by a small blower through the sampler. In typical vapor collections, the air flows at the rate of 0.5 to 0.75 liters/sec. At rates of this order of magnitude, the powder is constantly floating and is in excellent and uniform contact with flowing air.

During vapor collection the bed is kept at ambient temperatures, which are measured because the number of molecules collected through equilibration is a function of temperature. Experiments with n-butanol vapor in air indicated that at ambient temperatures the n-butanol content of the bed reaches two-thirds of the equilibrium value within 20 min and for all practical purposes reaches equilibrium value within 45 min. The rate-limiting factor is the diffusion of the organic material, e.g., n-butanol, into the greaselike film. Faster equilibration would require lower-viscosity films, which, however, would be more volatile and could introduce artifacts during analysis. Faster equilibration would also be achieved at a higher bed temperature, but a smaller finally collected amount and a loss of simplicity in collection would result.

Since collection involves equilibration, the actual airflow rate through the sampler does not have to be known and is not critical as long as the powder floats and does not pack at the end screens. Therefore it is unnecessary to measure airflow rates during sampling. However, water condensation in the sampler must absolutely be avoided, since condensed water constitutes a competitive collection phase having an unknown, variable volume. Also, if the concentration of the organic components in air changes rapidly during the sampling, the collected amount will not follow the trends well. Therefore the sampler is best suited for sampling from air in which the concentrations of the organics are reasonably

stable. The time constant of the sampler is of order of 20 min. Concentration fluctuations that are much slower or much faster than this value will be unimportant.

3.3 Vapor Transfer and Injection Apparatus

Vapor components trapped in the thin film in the fluidized bed are transferred, by elution with cryogenically purified helium gas (Figure 3), into a stainless steel thin-wall injector needle (Figure 4). During transfer, the bed material is heated to 80°C and the injector needle is cooled to liquid nitrogen temperature in a specially designed copper block immersed at its bottom end in liquid nitrogen. The typical duration of vapor transfer is 1 hr, and 70 to 80% of the organic vapor sample is transferred into the injector needle. Higher efficiency can be achieved by longer transfer at the expense of analytical time. Higher temperatures result in incipient breakdown of Apiezon L and peak artifacts.

The vapor sample in the injector needle is delivered to the chromatographic column by means of an injection system (Figure 5). The essential parts of the injection system include an electrical motor (A) with an eccentric cam, a stainless steel bellows (B), a stainless steel cylindrical bellows enclosure (C), a pressure gauge (D), a stainless steel capillary U-shaped cold trap (E), and a stainless steel injector needle (F).

The motor, acting through the eccentric and a spring-loaded pushing rod, can draw a 1-cm^3 volume of carrier gas from the injection port and into the bellows and later employ this volume to inject the sample from the heated injector needle into the septum of the column. The bellows enclosure provides a smooth fit of the bellows, with a minimum of dead space. The pressure gauge indicates the injection port pressure at any column temperature and any temporary pressure change due to injection. The gauge also serves to indicate, by a pressure lower than usual, any leaks in the septum/needle system. The U-shaped trap, which is 1/16-in

in outside diameter and cooled to liquid nitrogen temperature, blocks the passage of impurities from the bellows and the pressure gauge into the injector needle. When not in use, the injection system is kept under the flow of pure helium gas. During this standby interval, helium flows into the system at a measured rate (flowmeter G) and pressure (pressure gauge H) from a separate source through a liquid nitrogen trap (I) and a T-joint (K).

Sample injection involves the following steps. The needle containing the sample and kept in dry ice is attached to the injection system at the outlet of the cold trap by means of a Swagelok connector (L). The helium flow is then discontinued at K by closing the connection with a Swagelok nut provided with a metal rod. The other end of the injector needle is attached to the injection port of the chromatograph (M) by means of a Conax connector equipped with a Teflon sealing plug, and time is allowed for attainment of the injection-port pressure, as indicated by the pressure gauge (D).

A 1-cm^3 volume of carrier gas is drawn into the injection system by means of the motor-driven bellows. Again, time is allowed for stabilization of the pressure. To inject the vapor sample, the needle is heated to the selected temperature by passing a 20-amp current along the needle while the vapors are flushed into the septum with the 1-cm^3 volume of carrier gas stored previously in the bellows. The needle temperature rises from -80 to 180°C in 5 sec; it then remains at 180°C for the next 5 sec. Injection of helium gas from the bellows starts simultaneously with the heating of the needle and continues for 10 sec. During this interval, the helium from the bellows carries the vaporized sample into the septum while the normal carrier gas flow at $60\text{ cm}^3/\text{min}$ continues.

The injection system was tested on a large number of various samples, and it was found to produce reproducible and practically quantitative injections, with 95 to 98% of the sample introduced into the septum in one injection.

3.4 Calibration Apparatus

Calibration of the gas chromatographic readout for known compounds is relatively simple. To calibrate for any component of the vapor, the component must first be identified and obtained in a pure state from a commercial or a synthetic supply.* An airflow system (Figure 6) is set up in which breathing-quality air from a commercial pressurized tank is passed through an aerosol filter (A) of a material such as Teflon, a flowmeter (B), and a freshly activated carbon bed (C) cooled in a dry ice-propanol mixture (D) at its exit end. These precautions remove organic impurities from the air. The purified air is warmed to room temperature. It flows through a 2-liter three-neck flask (E), mounted with the necks facing downward, into a clean fluidized bed trap (F). One neck of the flask is connected to an upright small cylinder (G) with a small amount of pure component on its closed bottom. The cylinder is thermostated either at or below the temperature of the flask.

The amount of component that diffuses into the airstream in the flask depends on the vapor pressure of the component and the diffusion coefficient of the component in air. The diffusion rate can be approximately calculated from these values and from the cross section and the length of the gaseous diffusion path formed by the cylinder volume above the component sample. The amount is also directly measurable by experimentally determining the weight loss of component over a longer period of time.

With a controlled and known airflow rate through the flask and the sampler, the molecular concentration of the component in the air flowing through the sampler can be calculated. For instance, during calibration for n-butanol the source intensity

*For unknown components, their approximate concentration in air can be calculated if analysis is conducted using Apiezon L column, and the flame ionization detector's sensitivity is known; the gas chromatographic position gives the retention volume which can be recalculated to ambient temperature using Kovats Index system.

was 6×10^{15} molecules/sec and the airflow was $750 \text{ cm}^3/\text{sec}$. The concentration of n-butanol in the calibration stream was therefore 8×10^{12} molecules/ cm^3 . In a typical calibration this concentration of n-butanol in air resulted in an n-butanol peak with an area of 220 cm^2 at a sensitivity of 0.1×1 (the highest sensitivity setting on the chromatograph). This calibration procedure ties the concentration of a component in air directly to the size of the peak on the chromatogram if a standardized sampler and standardized sample-handling procedures are used. In the above case, 1 cm^2 of peak area at 0.1×1 sensitivity corresponds to 3.6×10^{10} molecules per cubic centimeter of air, and, since smaller peaks are detectable, the detection limit is somewhat lower than parts per billion by volume.

The factor relating the peak area to the concentration in air depends on the partition coefficient, k , for the component i in Apiezon L versus air; $k_i = (n_c)_i / (n_a)_i$ with n_a for concentration in air. It is possible to estimate the value of this coefficient from the amount of Apiezon L in the sampler, the estimated sample transfer efficiency during transfer from the sampler to the gas chromatograph in the standardized 1-hr elution procedure, and the knowledge (obtained by direct calibration) of the absolute number of molecules corresponding to 1 cm^2 of the peak area. For n-butanol in Apiezon L at room temperature, the partition coefficient was 4000. Therefore the described procedure corresponds to a concentration of n-butanol that is 4000-fold greater than that in air, and the analysis is equivalent to a one-shot injection of n-butanol contained in 4 liters of air.

If the molecular weight of Apiezon L is approximately 1000, the activity coefficient for n-butanol in this medium is, very approximately, 3. For larger polar molecules and for smaller but less-polar molecules, lower activity coefficients and correspondingly higher partition coefficients are expected. Table I shows calibration data for some polar and relatively small molecules for which Apiezon L is a poor concentrating medium.

Nevertheless, satisfactory collection is obtained even for these vapors. For the less volatile substances, a higher relative sensitivity is expected since the partition coefficient increases with decrease of the vapor pressure. Similarly, higher enrichment will occur with other, less polar vapors. When a vapor source is enclosed in a stream of air flowing at a known rate through the enclosure and then through the sampler, the total source strength can be calculated. For example, the rate of buta 1 production from a human subject was found in one case to reach 10^{15} molecules/sec.

3.5 Field Sampling Apparatus

For the collection of vapors emitted by humans enclosed in the glass cell (cf. Section 3.1) the fluidized bed sampler was placed at the air exit from the cell. It was possible to place two or three collectors in series and collect several comparable samples simultaneously.

For sampling in open environments, an apparatus shown in Figure 7 was used. It consisted of a 12-volt automobile type vacuum cleaner driven by a portable (airplane type) 12-volt battery. The cleaner pulled the air through the fluidized bed sampler. Provisions were available for water circulation through a glass jacket integral with the sampler, to maintain desired (room temperature) temperature level if the outside air was too cold or too warm. Since fluidized beds act as very efficient heat transfer media, additional pre-thermostating of air before entrance into the sampler would be necessary if ambient temperature extremes were encountered. In the present work, environment temperatures were selected so as to avoid such extremes and simplify the collection and calculations.

3.6 Gas Chromatographic Techniques

3.6.1 Packed Columns

The primary objective of the initial phase of the experimental work was the acquisition of a set of gas chromatographic patterns of human chemical signatures from subjects of various races and sexes as well as from different environments under simple and reproducible analytical conditions. For this purpose, a packed analytical column was selected and the analyses were performed under isothermal conditions at a moderate temperature. The selected column was 10-ft long and 1/8-in O.D. coiled stainless steel tubing packed with 20% Carbowax 20M supported on acid-washed 60/80 mesh Chromosorb P. During each signature analysis, the column was kept at 125°C in the air oven of the dual-channel Aerograph model 204 gas chromatograph. The selection of a packed column was based mainly on its relatively high stability or low rate of secondary aging - a favorable condition for minimizing the effect of column age on its analytical performance. The isothermal analytical conditions were chosen with some sacrifice on component resolution, to maintain maximum retention time reproducibility for each component of a complex mixture analyzed.

In the later stage of this investigation, two packed Apiezon L columns were also used in the three-column apparatus described below. In this apparatus, the packed columns were attached to one branch of the splitter connected to the outlet of a 200-ft open tubular Carbowax 20M column. The packed columns were selected to minimize the change in Apiezon L polarity due to the effluent impurities from the Carbowax column.

3.6.2 High-Resolution Column

In order to gain a deeper insight into the actual complexity of human chemical signatures, modern high separating power and high analytical speed columns were chosen and used in a

single column, a two-column, and a three-column gas chromatographic systems. The selected columns included a 50-ft, 0.020-in I.D. open tubular Carbowax 20M column, a 200-ft, 0.020-in I.D. open tubular Carbowax 20M column, a 50-ft, 0.020-in I.D. open tubular Apiezon L column and another 200-ft, 0.020 I.D. open tubular Carbowax 20M column, all support-coated.

The 50-ft Carbowax 20M column was used in combination with a mass spectrograph to help resolve human signature components in the attempts directed toward their identification.

The 200-ft Carbowax 20M column was used in combination with the 50-ft Apiezon L column in the two-column apparatus. The apparatus was designed to provide a primary resolution of sample components in the polar phase and a secondary resolution of the individual Carbowax 20M peaks in the non-polar phase. The two-column apparatus was used in characterization of chemical signature components obtained from the same Caucasian female. The apparatus is described in the following section of this report.

The second 200-ft Carbowax 20M column was incorporated into the three-column apparatus. In this arrangement, the split outlet of the long open tubular column was connected to two parallel short packed Apiezon L columns. The system was designed to produce simultaneously entire signature patterns in polar and non-polar phases and correlate the patterns by means of a specifically written computerized peak-matching procedure. The system was employed in the investigation of several signatures collected from a Caucasian female who also was the signature donor for the study performed on the two-column apparatus. The description of the system is given below.

3.6.3 Two-Column Apparatus

The modern high-resolution gas chromatographic columns provide a spectacular resolution of complex mixtures; however, many components still remain unresolved and they may be represented by single peaks. The two-column apparatus was designed and

constructed to effect resolution of such multi-component peaks and to provide data useful in characterization of the structure of the resolved components.

The two-column apparatus includes a 200-ft open tubular Carbowax 20M column (0.020-in I.D.), a 50-ft open tubular Apiezon L column (0.020-in I.D.), a 50-ft dummy column, a 3-way solenoid valve, two hydrogen flame ionization detectors and two sniffing ports. A schematic diagram of the arrangement of the apparatus is shown in Figure 8.

In dual column analyses, the Carbowax 20M column provides the primary sample resolution. The effluent from this column is split 1:1, and subsequently all or any part of the split effluent may be directed onto the Apiezon L column for secondary resolution. The peak switching step is effected by means of the solenoid valve. During this step, a switching blip appears on the chromatogram which denotes exactly the portion of the Carbowax 20M chromatogram transferred onto the Apiezon L column. Both analytical columns and the dummy column are provided with 10 cc/min helium flow. The 5 cc/min portion of the Carbowax 20M effluent is supplemented with 35 cc/min of helium and resplit, providing 20 cc/min flow to a flame detector and a sniffing port. The other 5 cc/min flow from the Carbowax column is mixed with 5 cc/min of helium and the total 10 cc/min is directed to either the Apiezon L column or the dummy column. At the exit of the Apiezon L column 30 cc/min of helium is added to the 10 cc/min flow and the total is again split so that 20 cc/min flows to the second detector and 20 cc/min to the second sniffing port. Approximately equal concentrations of sample reach the two sniffing ports; this permits a comparison of relative odor intensities when needed.

A segment of a typical gas chromatogram obtained in the dual-column analysis of a human chemical signature is shown in Figure 9. The upper portion of the figure represents peaks obtained on Carbowax 20M column; the peaks marked with asterisks were injected on Apiezon L column. The lower portion of the figure shows the

components of the injected peaks which were resolved on Apiezon L column.

The proper utilization of the two-column apparatus requires a knowledge of the retention dispersion characteristics for non-polar compounds as well as for selected polar compounds in Apiezon L stationary phase. The n-alkanes are expected to exhibit maximum retention on Apiezon L and the n-alcohols the minimum retardation. Other functional groups such as aldehydes and ketones are expected to fall between the retention times shown by the alkanes and the alcohols. The information on the retention behavior of selected compounds was acquired in a series of calibration experiments. In these experiments, the n-alkane series included C₈, C₁₀, C₁₁, C₁₂, C₁₃, C₁₄, and C₁₅ hydrocarbons, the alcohols were represented by ethanol, 1-propanol, 1-butanol and 1-pentanol; the aldehydes included propionaldehyde, butyraldehyde, hexanal, heptanal, and octanal; the ketone series embraced 2-butanone, 3-pentanone, 2-heptanone, and 2-octanone. The calibration experiments were performed under the same operating conditions and temperature (125°C) as used in the analysis of human samples. The Carbowax column effluent was allowed to flow continuously onto the Apiezon L column. For n-alkanes studied the experimental data were first expressed in terms of the respective elution times from both liquid phases; these data guided the experiments on human samples by providing the maximum time intervals required for a complete elution and display of all components of a given Carbowax 20M peak which was injected onto the Apiezon L column. The n-alkane data were then converted to corrected retention times and these were used in the construction of calibration curves needed in the characterization of components of human chemical signatures in terms of their respective Kovats Indexes on both stationary phases. The calibration data on n-alkanes are given in Table II.

The calibration data on polar compounds were first converted to respective corrected retention times. These retention times

were utilized in determining Kovats Retention Indexes for the selected compounds from previously obtained n-alkane calibration curves. The retention indexes were determined in Carbowax 20M and Apiezon L stationary phases. The respective Kovats Indexes were then employed in the construction of calibration curves for polar compounds which were displayed in the chemical logic field. These curves together with those constructed for other compounds from pertinent literature data served in the description of the structure of components of the human chemical signatures studied. The calibration data on polar compounds are summarized in Table III.

In order to minimize the inherent differences in composition of chemical signatures of different individuals, the collection experiments in this part of the work were limited to one subject and performed in a period of a few days. The selected subject was a young Caucasian female of high hygienic standards. During the experimental period, the subject was on an average diet. A total of four collections was made. In one collection, three fluidized Apiezon L beds were employed in series and two beds in series were used in each of the other collections. In the three-bed experiment, ten Carbowax 20M peaks were injected onto the Apiezon L column; in the remaining collections, eight, four, and three multi-component Carbowax 20M peaks were examined. The examination was performed with the two-column system kept at 125°C. The peaks observed on Apiezon L column and originating from the respective Carbowax peaks were described in terms of Kovats Retention Indexes and the data were plotted in two-dimensional (polar versus non-polar) chemical logic field to produce a tentative description of the structure of the components studied.

3.6.4 Three-Column Apparatus

The three-column gas chromatographic system was constructed to provide a detailed two-coordinate description (polar and non-

polar) of sample components from one single analytical run without the need for peak trapping and reinjection. The system is schematically depicted in Figure 10. The essential components of the system include a 200-ft open tubular Carbowax 20M column, a 6-ft x 1/8-in packed Apiezon L column, a 4-ft x 1/8-in packed Apiezon L column, and three flame ionization detectors. The outlet of the 200-ft column splits into two branches; one branch leads to the flame ionization detector, the other branch to two parallel Apiezon L columns. The outlet of each Apiezon L column connects to a flame ionization detector. Helium gas is delivered to the system from two independent flow trains. One train supplies the Carbowax 20M column with the flow rate of 5cc/min, the other train delivers helium to the Apiezon L columns; the flow rates are 40cc/min and 50cc/min in the long and the short columns respectively. The flame ionization detector residing at the outlet of the Carbowax column is supplemented with auxiliary helium.

The long, open-tubular Carbowax 20M column is used to provide the initial optimum sample separation. The separated sample components split into three fractions; two fractions simultaneously enter both Apiezon L columns; while, also at the same instant, the third fraction is detected by the detector attached to the Carbowax column. The Apiezon L columns are used to obtain the retention behavior for each sample component on a non-polar phase. The parallel arrangement of these columns and the combination of different lengths and carrier gas flow rates provide characteristic ratios of retention times which are useful in the subsequent peak matching procedure.

Each component of the sample is represented by three gas chromatographic peaks which emerge with characteristic sequence and elution times. The first peak appears on the detector receiving the signals from the Carbowax column, the second is eluted from the short Apiezon L column and the third from the long Apiezon L column. An example of a typical gas chromatogram (only a segment is given)

here) of a human chemical signature analyzed on three column apparatus is given in Figure 11. In order to extract useful information, the system is calibrated with various homologous series of compounds

In a typical calibration run a mixture of homologs and selected alkanes is injected into the system and analyzed at constant temperature. For each component of the mixture the characteristic elution times from all three columns are measured and converted to corrected retention data. The conversion requires the knowledge of retention characteristics for methane. This is obtained under similar analytical conditions by incorporating the component into the sample or analyzing methane before injecting the sample into the mixture.

The corrected retention data are used in the establishing of the peak shifts which occur on the Apiezon L columns. These shifts are given as the ratios of the corrected retention time for each component on the long Apiezon L column and the short Apiezon L column. The value of these ratios which was found to be constant for all compounds studied is used in the peak matching procedure in the interpretation of the unknown samples. The corrected retention data are also utilized in the computation of the Kovats Retention Indexes for the components of homologous series in the two stationary phases employed. The retention indexes serve to construct calibration curves which are displayed in a chemical logic field for final characterization of components of actual samples.

The results of experiments on calibration of the three column apparatus are summarized in Tables IV to X. The data on calibration performed using n-alkanes with the three column system kept at 90°C, 120°C, and 150°C are given in Tables IV to VII.

It is noted that the ratios of the corrected retention times observed on Apiezon L column for all compounds studied are practically constant and temperature independent. The average

value of these ratios was used in the computerized indexing of the human chemical signature components.

The results of calibration experiments carried out with polar compounds are summarized in Tables VIII to X. The ratios of the corrected retention times on Apiezon L columns for all compounds studied are also constant and temperature independent. The elution data listed in these tables were used in the three-point match procedure designed to inspect the three gas chromatographic records for the presence of such compounds in human chemical signatures.

3.6.5 Calibration for Conversion to Kovats Indexes

The initial series of gas chromatographic patterns of human chemical signatures was acquired without calibrating the analytical system with a synthetic mixture of n-alkanes. In order to evaluate the daily instrumental variations affecting the positions of the signature components in their respective patterns and to assign Kovats Indexes to those components, a set of two peaks always observed in each pattern was selected and used as pattern "internal standard". One of the selected peaks emerged in the initial section of the patterns with an average retention time of 2.80 ± 0.09 min. (corresponding to n-butanol), the other peak was observed in the final section of the patterns with an average retention time of 27.84 ± 0.47 min. The results of the retention time standardization for the initial series of the chemical signature patterns were then correlated with those obtained in the second series of patterns which were standardized with a synthetic mixture containing hexane, octane, decane, dodecane, tetradecane, hexadecane, and 1-butanol. The correlation facilitated the assignment of the statistical pattern analyses of both series of patterns and provided data needed in the computation of Kovats Indexes for all pattern components.

The Kovats retention indexes were computed for each component of the signatures by using the following equation:

$$I(A) = 100 \cdot (y-x) \frac{\log (t_{ra}/t_{rx})}{\log (t_{ry}/t_{rx})} + (100 \cdot x)$$

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where

$I(A)$ = retention index of compound A
 t_{ra} = retention time of compound A
 t_{rx} = retention time of n-alkane $C_xH_{2x} + 2$
 t_{ry} = retention time of n-alkane $C_yH_{2y} + 2$

For most of the components the calculations were made by interpolating for

$$t_{rx} \sim t_{ra} \sim t_{ry}$$

Only a few values were obtained by extrapolation. To satisfactorily determine the absolute retention values needed in the computation of the Kovats Indexes, it is necessary to know the gas hold-up time of the column under standardized analytical conditions. Since it is impossible to measure this value if the detector is insensible to inert gases, the retention time for methane is usually taken as equal to the gas hold-up time. The value was measured (0.31 min) and used to correct the retention times of the components of the human signature and synthetic mixture.

The patterns of human signatures were then compared with those of various environments and Kovats Index ranges for Carbowax 20M peaks were selected which were most significant for discrimination of humans and environments.

In order to obtain a more precise characterization of the most significant components of signatures, a systematization process was introduced to treat the data obtained on the high-resolution two-column apparatus. The principle underlying the process is illustrated in Figure 12.

The X-axis is the Kovats Index scale for retention in the Carbowax 20M column. The Y-axis is the delay, in minutes, in the Apiezon L (second stage) column, after correction for hold-up time which was obtained by calibration with methane. The Kovats Index scale that corresponds to these delays is shown on the right. The upper diagonal line represents the delay for the n-alkanes and other non-polar saturated hydrocarbons. This is then the maximum delay which a component with a given Kovats Index in Carbowax 20M can

experience in the non-polar Apiezon L column. If the compound is somewhat polar, or possesses double bonds, it will be delayed less and its point in the two-index field of Figure 12 will be lower. The zones where various types of compounds reside, on the basis of literature data, are indicated. Straight chain alcohols are the most polar of the compounds considered here (the glycol zone is also shown) and are located on a straight line. This is drawn using our own experimental points. Points for some other compounds not reported in literature are also shown.

Branched alcohols, or alcohols with OH groups not on the terminal carbon atom lie above the n-alcohol line, but short of the carbonyl compound zone. Double bonds e.g., in the alcohols, bring the points to a zone somewhat below the non-double bond compounds of the same type. Notice that the point for n-butanol lies in the alcohol zone of the continuation of the line formed by higher n-alcohols and at the same horizontal and vertical distance from hexanol. In this particular case, only a rather unusual compound (e.g., some carbonyl compound with more than one double bond) has an outside chance to produce a point that might be confused with n-butanol.

A set of calibration compounds was selected to permit frequent calibration of a plot of Figure 12 for several homologous series. This is desired to increase precision and reliability, since although the plot as it is represented appears to be reliable within ± 10 K.I. units, the actual columns used can differ slightly from the performance of columns on the basis of which Figure 12 was drawn. The causes for difference are slight differences in the average molecular weight of Apiezon L, slight oxidation, contribution of adsorption at column walls, etc.

3.7 Mass Spectrographic Identification Attempts

Various attempts were made to identify components of human chemical signature by mass spectrography. The initial experiments

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were performed on signatures collected from a Caucasian male and from an Indian male. In these experiments, the packed analytical Carbowax 20M column was connected to the Hitachi Perkin-Elmer model RMU-6D mass spectrometer with a Watson Bieman sample enrichment device. The column effluent with human signature components was split so that 10cc/min entered the ion chamber and 50cc/min the flame ionization detector. The analysis was performed at 125°C.

Another series of experiments was designed to evaluate the minimum amount of component needed for its positive mass spectrographic identification to collect larger human samples and to improve its resolution. For this purpose, 100-g Apiezon L fluidized bed was constructed and tested in an experiment on signature collection from an Indian male. To improve the component resolution and to facilitate the interpretation of mass spectrographic data, a 50-ft x 0.020-in I.D. support-coated open tubular column with Carbowax 20M as the liquid phase was employed. The column effluent was fed directly into the ion chamber at the rate of 10cc/min. During the analysis, the column temperature was programmed at 2°/min from 60-180°C. The peaks were detected on picnoammeter and timed from the instant of sample injection to the appearance of the maximum current for each peak investigated. In experiments with standard-size beds, each mass spectrographic analysis was preceded by gas chromatographic analysis of human sample collected from the same individual. This was effected by collecting samples on several beds in series. The samples were collected from a young Caucasian female.

The minimum amount of compound needed for positive mass spectrographic identification was estimated with a mixture of 1-butanol and heptanal. For this purpose, various aliquots of the mixture were analyzed under the same conditions as used for human sample. The aliquots were selected to match major and medium size peaks obtained from human sample. In each case, attempts were made to identify the selected compounds by mass spectrography.

3.8 Statistical Pattern Analyses

As the gas chromatographic data on chemical signatures of humans obtained by the fluidized bed collector technique began to accumulate, it became evident that the composition of the organic vapors from humans varied significantly from individual to individual. Visual inspection of the chromatograms from males and females did not indicate obvious common patterning (except e.g., such as a peak in that location where n-butanol should occur) nor obvious group differences. At that stage of work, patterns consisted of 32 to 48 peaks; the sizes of the peaks in the same gas chromatographic location varied within a wide range from individual to individual, and a comparison of any two gas chromatograms indicated that always a fraction of peaks could be approximately matched with respect to their positions on the gas chromatograms, while others would not match.

The factors that could explain these experimental findings were several. Some did not become apparent until high-resolution two-column gas chromatography was employed toward the end of the project work: higher resolution chromatography indicated that 60 to 100 peaks could be observed, and the two-column apparatus (Section 3.6.3) demonstrated that most of these still could be further resolved into 2 to 10 peaks, each corresponding to at least one organic component. Hence, the 32 to 48 peaks observed in analysis in packed column (Section 3.6.1) each corresponded to mixtures of several components, close in their retention times, and the position of the compound peak would be expected to shift as the proportion of the components in the compound peak would change from the individual to individual. Other causes of the peak position shifts may be the usual experimental fluctuations in the retention times, and the presence of some water in the samples. The amount of water in each injected sample was estimated at a few milligrams. The hydrogen flame ionization detector will not respond to water vapor to a significant extent, but in the Carbowax 20M analytical column the water, while

passing through the column, can temporarily increase the polarity of this stationary phase and can for short time intervals, influence the rate of migration of the components through the column. The retention times of the less polar components will slightly decrease while those of the polar components will increase.

Because of the large variety of the gas chromatographic features observed, it became necessary to develop criteria for selection of those features that would be most significant for identification and classification of human chemical signatures. At this juncture, a series of statistical pattern analysis methods were applied. In the present section, the reasons for use of the different methods will be briefly outlined; the procedures and the results are discussed in Section 4.2 and its subsections.

Procedure A

Various similarity indexes have been proposed in literature for comparisons of patterns or stimuli. In general, the indexes attempt to compare the extent of those features which are similar with those which are dissimilar, or with the total input of features. The crucial part of the procedure is the decision making on the correspondence of features. In the case of the gas chromatographic patterns, the positions of the peaks in two gas chromatograms may either match, or mismatch to varying degrees. The decision must be made as to how much mismatch can be allowed while still considering that one deals with the same feature in both gas chromatograms. A computer program was developed, designated GASCHR to compare each peak in each chromatogram with peaks in the other gas chromatograms with prescribed values of tolerance (typically 4 percent in retention time) in mismatch. The peak matching requires certain positive or negative strain if the match is not exact, and the total algebraic sum of the strain over those peaks which were matched was calculated and printed out. Usually, when approximately the same numbers and amounts of the negative were required, the sum would not differ too much from zero. Some

peaks were matchable, within the prescribed tolerances, to two peaks in the other gas chromatogram.

The peak matching generated, for each pair of patterns, a basis for calculating the various similarity indexes:

Tanimoto Index*. This similarity index is +1 when the two patterns match exactly, and zero when the patterns do not match at all (wherever there is a peak in one pattern, there is none in the other).

The Tanimoto index is calculated as follows: After matching the two patterns within some prescribed tolerance limits (e.g., in one variant of our program, peaks whose retention times in the two patterns differed less than by 4 percent were considered matched and forming an "identical pair"; if one peak in one pattern had several candidate matching peaks, within the chosen tolerance limit, in the other pattern, the peak which matched best was chosen as the partner of the matching pair), sets of peak pairs are taken to describe the correspondence between the patterns. Those peaks of the first pattern which had no match in the second pattern, or in reverse, formed pairs in which one partner was zero. The Tanimoto index S_T is the ratio of the sum of the smaller partners of the pairs divided by the sum of the larger partners of the pairs. A derivative measure of the similarity is Tanimoto "distance" between the patterns

$$D_T = -\ln_2 S_T$$

*Rogers, D.J. and Tanimoto, T.T., "A Computer Program for Classifying Plants," Science 132, 1115, 1960.

Tanimoto, T.T., "Nonlinear Model for a Computer-Assisted Medical Diagnostics Procedure," Trans. N.Y. Acad. Sci. Ser. 2, 23, 576, 1961.

Tanimoto, T.T., "A Class of Exponential Distributions and their Associated Minkowski Geometries," Notices Am. Math. Soc. 8, 432, 1961.

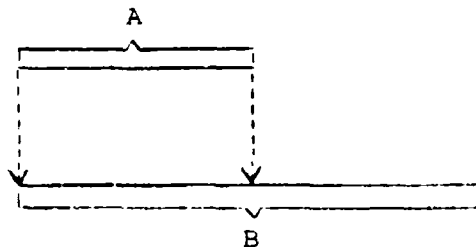
Ornstein, L., "Computer Learning and the Scientific Method: A Proposed Solution to the Informational Theoretical Problem of Meaning," J. Mt. Sinai Hosp. 32, 437, 1965.

It is zero when the patterns are identical, and infinite when the patterns completely mismatch. One of the advantages of this index is its ease of computation and a lack of bias in favor of large differences in one or a few features such as exist in the similarity indexes using quadratic terms. Since the large differences in a few features cannot a priori be assumed significant in pattern classification, it is undesirable to overemphasize their influence in forming the similarity index.

Ekman's Index. Mathematically, it is a simple function of Tanimoto index, but it is closer to the psychophysical concept of similarity presumably operative in the comparison of two stimuli by humans and developed independently of Tanimoto. Ekman and Eisler* consider that the similarity concept follows a process that is mathematically equivalent to taking the sum of those parts of attributes common to both stimuli (here we consider patterns as "stimuli") and dividing this sum by the sum of both full stimuli. Thus, Ekman's similarity index for two lengths A and B is:

$$S_E = \frac{A + A}{A + B}$$

where



This simple concept apparently suffices for reasonably good stimuli recognition by human senses.

Taking then, a bionic approach, we adapted Ekman's concept for the comparison of two patterns through the following procedure:

*G. Ekman, "Some Aspects of Psychophysical Research in Sensory Communications," Rosenlith, ed., MIT Press, 1961, p. 35.

1. Each pattern is first normalized within itself.
2. "Corresponding" peaks within both patterns are indexed by using the 4% rule. This leaves surplus peaks in both patterns. The result is a set of pairs of attributes. For those peaks that have no corresponding peak in the other pattern, the corresponding peak has zero intensity.
3. The normalized intensities of each pair of corresponding peaks are written side by side.
4. The smallest sides of each of the corresponding peaks are added; the sum is multiplied by two.
5. All normalized intensities for both patterns are added.
6. The result of step 4 is divided by that of step 5, and gives S_E (Ekman's similarity index). It will be small (approaching zero) if the similarity is small; it will be +1 for exactly similar patterns.
7. The similarity index, S_E , is converted to a distance value by taking $-\log_2 S_E$. For identical patterns, the "distance" is zero. For highly dissimilar patterns, the distance, D_E , is a positive number exceeding 1, 2, or more.

Correlation Coefficient.

This is the well-known classical measure of correlation between two sets of observations, or, in the case of pattern comparisons, of two patterns at a time, aligned so that the peaks match in their retention times within some prescribed tolerance limits.

The classical correlation coefficient (very related = +1; unrelated = 0; oppositely related = -1) also gives intuitively correct values for distances between points in the correlation space, but is more difficult to compute. Also, it can give a value of +1 for patterns in which additive and multiplicative differences compensate, while patterns are intuitively dissimilar.

In the statistical experiments with the similarity indexes, additional variations were: Tanimoto index averaged over peaks, Ekman index summed over and divided by the number of pairs. To minimize the influence of the quadratics of the correlation coefficient, a correlation coefficient applied to normalized area X transformed logarithmically

$$y = \log (x + 0.02)$$

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was included, in addition to the correlation coefficient applied directly to the normalized areas*.

The rationale behind the study of the similarity indexes was the possibility of developing a taxonomy (classification) of the patterns. If a third pattern has high similarity index to each of the two patterns which also have a high similarity index, these three belong to the same class. Otherwise, a new class can be set up and each next pattern tested for belonging to one or the other class etc. Classes can also branch, cf. Figure 13. Here pattern A has some similarity to B (2 of 5 features match, $S_T = 0.4$), C has more similarity to B (3 of 5 features match, $S_T = 0.6$) but A and C are completely dissimilar (none of the features match)**. Thus interrelation between classes can be gradually developed, and basically must use non-Euclidian spaces for hypergeometric representations (in Euclidian space, if A is close to B and C is close to B, A and B cannot be infinitely far apart).

Although the taxonomy development using similarity indexes indicated that vapors from similar sources in average tended to produce patterns with higher similarity indexes, the indexes per se were low indicating that much extraneous noise (non-essential features) interfered with the classifications. Therefore other methods were applied.

Procedure B

In this procedure, a more mechanical approach was taken to reduce the patterns to a definite number of variables to which

*During this stage of work, the normalized areas were taken as the areas divided by the mean area of peaks in the particular pattern. In later work, second part of Procedure D, the normalization was by dividing the peak areas by the total area of all peaks in the respective pattern.

**Tanimoto distance between Tanimoto units B and C is $-\ln_2 (0.6) = 0.73$.

then stepwise discriminant analysis program was applied. The chromatograms (initially in terms of retention times normalized to n-butanol, later in terms of Kovats Indexes) were divided into ranges of equal width, (or percentage width), disregarding the initial and the last portion of the gas chromatograms. Peaks with the position of the peak in the particular interval were considered to be, with their full area totally in this interval. If several peaks occurred in the same range, their areas were summed and the sum became the intensity coefficient for this range in the respective gas chromatogram. Thus, a "variable" was the total content of organic substances eluted during the particular range, with peaks in this range. By increasing the width of the ranges, the number of range boundaries could be decreased, reducing the number of cases when the same substance eluted close to the boundary could occur in one or the adjoining cell.

After reduction of the chromatograms to a set of number of variables stepwise discriminant analysis was conducted, using BMD07M Stepwise Discriminant Analysis Computer Program, Version of September 1, 1965, Health Sciences Computing Facility, University of California, Los Angeles.

Procedure C

This procedure also used stepwise discriminant analysis, but instead of selecting the variables by mechanical subdivision into ranges, a histogram study was conducted to find those gas chromatographic locations (in terms of Kovats Indexes) in which peaks most frequently occurred in the chemical signatures of humans. The variables were then formed by putting the center of the range at the maximum frequency point and allowing ± 10 Kovats Index units as an estimated tolerance for the two boundaries of the same range. Thus, in this procedure, the variables were histogram-guided ranges, not necessarily with common boundaries scattered throughout the gas chromatograms. Again, areas of all peaks with the apex in the range formed the intensity value for

the particular variable.

Procedure D

This procedure was a further development of the Procedure C, and differed simply by introduction, into the stepwise discriminant analysis, of the "non-peak" ranges, between the ranges where peaks were frequently observed in humans.

The above various procedures were applied to all peaks, or, in some trials, only to peaks within certain peak size classes, (e.g., only large peaks, above certain size, etc.)

The stepwise discriminant analysis looks for those features which are most significant in discriminating two or more groups of patterns, and forms discriminant functions which are linear equations of the form:

$$X = a_1 V_1 + a_k V_k + \dots$$

Here a_1, a_k etc. are coefficients derived by the computer program, while V_1, V_k etc. are the gas chromatographic variables. The V 's were not taken as the direct sums of peak areas within the particular gas chromatographic range but were logarithmic transforms for the corresponding areas, so as to give more equitable weights to large and small peaks. In the Procedure D, the transform was, e.g.,

$$V_{34} = \sum_{1112}^{1132} \left[1 + \log (A_i + 0.1) \right]$$

where 34 is the "name" of the range; 1112 and 1132 are its boundaries in terms of Kovats Indexes; A_i is the peak area of a peak within this range, taken in terms of its area as percent of the sum of areas of all peaks in the same pattern. If i was the only peak in the range, the expression in the square brackets became V_{34} . If there were several peaks in the range, V_{34} was the sum, as indicated, of the logarithmically transformed peak areas.

Discrimination means that a function X , or several functions (X,Y) are found such that their values for all patterns of one class, (e.g., humans) are distinctly different from the values for another class (environments).

These functions can then be used to discriminate one group of patterns from another. The same principle applies to discrimination of several groups of patterns.

In the stepwise discriminant analysis the success of the procedure can be judged by two criteria. If the number of variables used is small compared with the number of patterns used (much less than by a factor of 2), and function that separates the patterns in two (or more) groups is found, the probability that this is a valid discriminant function is high. The probability increases as the number of patterns is increased and the number of variables needed to classify decreases.

The other criterion is more rigorous. If the discriminant function is obtained by using only a part, e.g., one half of all of the patterns (each pattern used fully), its validity can be tested using the values of variables from the other half of the patterns. If the other half of the patterns is satisfactorily classified into its proper group, the probability is high that the variables selected by the discriminant analysis constitute truly differentiating pattern class features.

3.9 Subject Selection and Preparation

The signature collection experiments were performed on human subjects of both sexes and various races. Except for two middle-age Caucasian males, all subjects were young, healthy specimens. Before the participation in the experiments, the subjects did not use any cosmetic preparations except for neutral soap, and lived on normal mixed diets. To expose the maximum area of skin to air flow the subjects were closed in the cleaned glass system partially naked.

The selected human races included American Caucasian males

and females, Indian males, American Negro subjects, and Mexican and Malayan subjects.

The majority of the Caucasian males were students at the Illinois Institute of Technology in Chicago. The student group included two pairs of brothers. The rest of the Caucasian males comprised two chemists, one free-lance writer and one chemical laboratory technician.

The group of Caucasian females was composed of young office workers employed at IIT Research Institute, house-wives and a few high-school students. This group included a mother and her two daughters.

The Indian group consisted of young males born in India with temporary residence in the United States. All members of this group were students at the Illinois Institute of Technology in Chicago.

The American Negro group included one young male chemist and two young females. The male subject was a husband of one of the females.

The last group included a young Mexican house-wife born in the United States and a young Malayan male. The male subject was also a student at the Illinois Institute of Technology in Chicago.

Other sources of chemical signatures consisted of kitchen and laboratory air and tropical vegetation. The collection of these signatures were performed by means of the portable air sampling apparatus. The kitchen air samples were obtained from the IIT Research Institute Cafeteria kitchen; also the laboratory samples and machine shop samples were taken at IITRI.

The chemical signatures from tropical vegetation were collected in the Palm House of the Lincoln Park Conservatory in Chicago. The collection environment included various types of palm trees, such as sentry palms (Howea forsteriana); fishtail palms, a native of India and the Malay region, (Caryota mitis); and scattered through the underplanting of Fittonia, a

number of ornamental palms, such as Fiji Island fan palms (Eupritchardia pacifica), silver palms (Coccothrinax argentea), and roebelen palms (Phoenix roebelen). There is also a 50-ft fiddleleaf rubber tree (Ficus lyrata) from Africa, a tapioca plant (Mauihot exculenta), a fig tree (Ficus carica), and the common banana (Musa sapientum). The Conservatory also houses flowerless plants such as ferns and mosses. The temperature and the humidity of the Palm House environment range from 60 to 85°F and from 50 to 75%, respectively.

4.0 RESULTS AND DISCUSSION

4.1 Packed Column Data

The experimental data on human chemical signatures of both sexes and various races as well as of different environments are tabulated in the form of copies of computer printout in Appendix I.

In the tabulation, each pattern is identified by the proper number, the signature source ("subject": initials of humans), the date of signature collection, the total intensity and the mean peak intensity.

The arrangement of patterns follows approximately the chronological order of signature collection experiments and is numbered accordingly.

The total intensity is defined as the total area, in sq. mm., of all gas chromatographic peaks of the particular pattern and the mean peak intensity as the total intensity divided by the number of peaks in the same pattern.

The data on each pattern tabulated in Appendix I includes also consecutive numbers of peaks in the pattern, the retention times of peaks in terms of Kovats indices, the peak areas in sq. mm. (calculated to the electrometer sensitivity setting of 0.1×1) and the peak normalized areas (peak area divided by the total intensity).

The gas chromatographic patterns of the signatures studied reflect complex and only partially resolved mixtures. The number of detectable components observed in human signature patterns ranged from 32 to 48 with many indication for the presence of a large number of additional unresolved peaks. The magnitude of the collection samples ranged from $2.6 \times 10^{-7}g$ for an Indian male to $2.1 \times 10^{-5}g$ for a Caucasian male.

The total number of detectable components in the environment chemical signatures ranged from 34 to 46 with a strong evidence for the presence of many unresolved peaks. The magnitudes of the collected samples fell within the range of $1.9 \times 10^{-6}g$ and $1.1 \times 10^{-5}g$.

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It must be understood that each peak in the tabulation can represent more than one substance and is resolvable, as later work with the two-column device showed, into several peaks when examined in a non-polar Apiezon L column.

The peak areas are approximately proportional to the sum of weight of the components represented by the peak. Table XI lists the relative amount of area contributed per atom of the respective element*.

Since the number of heteroatoms compared with the number of carbon atoms in common compounds with reasonable molecular size is small, the area of multicomponent peaks can be assumed to be approximately proportional to the weight of material it represents.

The peak areas in the Appendix are quoted in mm^2 , taken at the electrometer attenuation of 0.1×1 . For the particular instrument, the calibration factor was approximately:

$$1 \text{ mm}^2 = 0.2 \times 10^{-12} \text{g}$$

The peak areas represent the amount of the organic material within the peak range injected into the chromatograph. The amount present in the collected sample is estimated, from the efficiencies of sample transfer and injection to be 30 percent higher.

The concentration of the respective materials in air from humans could be calculated only if the calibration factor (partition coefficient) was available for the particular molecular species: Table XII represents data on the emission rates of n-butanol from humans. This substance formed a recognizable peak used for internal calibration.

*Johns & Sternberg, Gas Chromatographic Detectors, in Instrumentation in Gas Chromatography, p. 179, Krugers, ed., Centrex Publishing Co., Eindhoven, Holland, 1968.

For unknown substances, very approximate calibration is possible provided that Kovats Index of the unknowns in Apiezon L is known and taken to be, at room collection temperature, the same as at elevated temperatures. The amount G of a substance X collected is

$$G = k \cdot m \cdot v$$

Here k is the partition coefficient of the substance in Apiezon L/Air system at the collection temperature (ambient); m is the concentration, in grams/cm³, of the substance in air, and v is the volume of Apiezon L in the collector. A plot of log k versus the Kovats Indexes of the substances in Apiezon L is a straight line and can be used for approximate estimates of k; v is known, and m can be calculated when G is known from the area of the gas chromatographic peak. The value of k increases by a factor of approximately 2.3 per each 100 units of Kovats Index increase. The emission from the human subject is (750 ...)/sec when 750 cm³/sec is the rate of airflow through the glass cell from which the human subject vapors are collected.

These remarks are to indicate that in the equilibration sampling (fluidized bed, Section 3.2) approximate calibrations can be obtained even for unknown materials, from their Kovats Index positions in Apiezon L. Since the Apiezon L Kovats Indexes were obtained in the two-column device (Sections 3.6.3 and 4.4) where the first column was a 200-ft open tubular Carbowax 20M column, the areas under peak in the packed Carbowax 20M column cannot be directly related to the high-resolution runs which resulted from the use of two-column device. The packed column analyses were to assist in finding those gas chromatographic regions where the significant features reside. It was more important to concentrate adequately all organic vapors including those with a low volatility, so that they would be detectable at feasible sensitivities and to analyze these in Carbowax 20M column where better peak shapes are obtained for all polar components, including alcohols which chromatograph

poorly on Apiezon L. The use of Carbowax 20M in the fluidized bed collectors was not practical since the sample collected too much water which then interfered with the analysis.

4.2. Pattern Classification

The various approaches to this task were discussed in Section 3.8. The objective of the pattern classification was twofold: (1) to obtain evidence that the complex chemical signatures, in the form of gas chromatograms of vapors from humans and environments, contain features useful for their identification as to the type of their sources, and (2) to indicate those regions in the gas chromatograms that are most significant for the source discrimination and therefore merit first attention in further chemical identification of the components.

4.2.1 Peak Matching Studies

Some pattern comparisons were conducted using simple rank order statistics on gross indexes such as the number of peaks per chemical signature. Here three more or less homogeneous groups of humans on which a sufficient number of chemical signatures were available were used: 12 males from India, 25 Caucasian American males and 23 Caucasian American females.

The average numbers of peaks and the standard deviations in the numbers of peaks (in parentheses) in the chromatograms submitted to the peak matching procedures were as follows: Indian males 41.2 (1.8); Caucasian males 37.3 (2.5); Caucasian females 40.0 (4.2); environments 37.0 (6.1). Wilcoxon-White rank order test applied to the number of peaks per gas chromatogram as the ranking criterion reached 99 percent confidence level (less than 0.01 probability to obtain such differences by chance) when Indian males were compared vs Caucasian males, and when Caucasian males were compared vs Caucasian females; the differences in the peak numbers between Caucasian females and Indian males did not reach 95 percent confidence level.

Further comparisons included data inspection after peak matching.

Most of the pattern comparisons for the purpose of obtaining similarity indexes were conducted after matching of peaks between two patterns at a time, allowing for a ± 4 percent tolerance of retention time normalized to n-butanol after addition of 0.02 to the normalized retention time. The 0.02 term was significant only at the initial time range of the gas chromatographic patterns.

The selected matching criterion was apparently rather liberal, since the number of peaks that could be matched within each pair of chemical signatures taken was in excess of 80 percent of peaks observed in each of the patterns of the pair. To assess if at this tolerance level gross differences existed between members of the same group versus the numbers of the same or other groups, seven randomly selected patterns were chosen within each of the three groups and compared with others. The following observations resulted: from the average number of 37 peaks in 7 Caucasian male patterns, 85 percent found matches in the patterns of other Caucasian males; from the average number of 40.7 peaks in 7 Indian male patterns, 82 percent found matches in the patterns of Caucasian males; from the average number of 39.6 peaks in 7 Caucasian females, 84 percent found matches in the patterns of other Caucasian females. These initial observations did not indicate existence of gross differences in the match frequencies within the group and between the groups.

The relatively high density of the coverage of the gas chromatograms with peaks is one of the factors which may account for this initial result. If the allowed match tolerance is too broad, there will always a high probability of finding a matching peak. If the tolerance is too narrow, experimental variability of the retention times for the identical substance may be large enough to miss the tolerance limit and give "no match" although actually a perfect chemical identity match existed.

Consequently, more elaborate pattern similarity indexes were explored in which consideration was given not only to match versus nonmatch in the peak positions but also to the magnitudes of the chromatographic areas which represent the abundancies of the components eluted in the respective gas chromatographic range.

The merits of two similarity measures--Tanimoto distance and the correlation coefficient (cf. Section 3.8)--applied to patterns matched to 4 percent tolerances were explored on a part of the data available at the time when the computerized GASCHR program became operational.

The Tanimoto distance in a non-parametric hyperspace is zero for two identical patterns, large for dissimilar, and infinite for completely non-matching patterns in which no peaks in one match the other pattern's peaks in retention times within prescribed tolerances.

The test group consisted of the chemical signatures of 12 Indian males, 10 Caucasian females, and 9 environments, (2 plants, 2 laboratories, and 5 kitchens). The results indicated that considerably variations occurred within each group and that very few Tanimoto distances were below 1.0. However, the average distances for pairs within each group and for intergroup pairs were consistent, and patterns for humans were clustered, in comparison to the average Tanimoto distances between chemical signatures of humans and environments.

Indian males versus Indian males	1.561	(66 pairs)
Caucasian females versus Caucasian females	1.817	(45 pairs)
Indian males versus Caucasian females	1.809	(120 pairs)
Indian males versus environments	2.094	(108 pairs)
Caucasian females versus environments	2.024	(90 pairs)

This tabulation shows that (1) the Indian male group was most consistent within itself (2) the variability among Caucasian females was larger and approximately as large as between Indian males and Caucasian females, and (3) the difference between human and the environmental chemical signatures was larger.

Correlation coefficients applied to the logarithmically transformed peak areas after 4 percent tolerance match showed

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similar relations. The largest cohesivity occurred within the Indian male group.

When patterns were assigned to human versus environment groups by comparing the average Tanimoto distances from the pattern in question to 26 human patterns and to 9 environment patterns correct assignment resulted. Pattern assignment to certain groups of humans on the same comparison basis was inconsistent. It was concluded that if the Tanimoto distance or a similar concept is used, the number of comparison samples should be increased, or some pre-classification of peaks into useful and nonuseful should be conducted.

The similarity indexes for patterns within the same group were relatively small - 0.3 to 0.5, rarely reaching above 0.5. Many mismatches in sizes and appearances of spurious peaks corresponding to occasionally occurring components in vapors from humans lower the index with the result that many Tanimoto distances were above unity and were not good classifiers.

Thus, the pattern classification approaches based on similarity indexes did indicate the possibility of classification. However, it was felt that discriminant analyses where the emphasis is not on the measures of similarity but on the features most useful in class separations may be a more powerful tool for the cases when the classes to which the various patterns belonged were already defined in terms of the vapor sources. Similarity indexes are more specifically suited for developing taxonomies when the observations are not easily assignable to classes, but classes and subclasses have to be uncovered in poorly structured data.

Consequently, later work was concentrated primarily on using discriminant analysis as the classification tool.

4.2.2 Stepwise Discriminant Analyses-Arbitrary Variables

For the stepwise discriminant analyses (cf. Section 3.8) when the chemical identity of the components is uncertain it is

necessary to subdivide the gas chromatographic patterns into a fixed number of ranges serving as variables (vectors), so that each pattern can be expressed in terms of the same number of variables. If each range contains not more than one peak, the intensity value for the particular variable can be the normalized area, or some function of it, of the peak. Experience indicated that if the ranges are small enough to contain either none or very infrequently more than one peak, many cross-overs can occur in a certain range, or in the neighboring range while representing the same component, since the position of a peak can shift by several reasons (cf. Section 3.8).

Initially, 65 ranges were set up, with the width of each range corresponding to 6 percent of the retention time corresponding to the ranges lower retention time boundary; e.g., for the range beginning at 10.0 minutes the boundaries would be 10.0 and 10.6 minutes, respectively. This partitioning resulted in 65 ranges. After preliminary runs, with 49 patterns available at that time in which the retention times were normalized with respect to the n-butanol retention times, 21 adjoining (4 with retention times smaller than that of butanol and 16 with retention times larger than that including the butanol) were selected, since (1) the number of patterns presented for analysis must exceed the number of variables, (2) the retention times of peaks around the butanol peak were more reliable when relative to butanol, and (3) preliminary analysis showed that the majority of peaks useful for distinguishing various types of humans seemed to be in this region.

By using a correlation coefficient matrix, the statistical process extracted successive significant variables, beginning with those which produce the most efficient separation between prescribed groups of patterns. It continued to extract the variables (peaks) in order of their decreasing significance. After each variable had been handled for all patterns, the efficiency of classification based on all peaks extracted up to this point

was evaluated. This process was applied to the 49 patterns grouped in 5 groups: 20 Caucasian males, 12 Indian males, 10 Caucasian females, 5 various humans of other types, and 2 tropical plants.

To compare the efficiency of the classification process by these logical groups, a random classification of the same 49 patterns was also subjected to analysis. Figure 14 shows that after classifying 13 variables, only 3 patterns, or less than 9%, were misclassified when proper groups were used and that 15 patterns, or over 30%, were misclassified in the randomized groups. The partial classification structure in the randomized group arises from the statistical probability that a pattern can be assigned to the proper groups by chance and from the presence of possible other classifiable features that can occur in the gas chromatograms.

Subsequently, the range of the gas chromatogram included in patterns for the stepwise discriminant analysis was expanded and the width of each range representing separate variables was doubled. The purpose of this change was to include more information and reduce the probability, for the peaks close to a range's boundary, to cross over the boundary into the adjoining cell. The range of retention times between 0.327 and 5.73 (relative to n-butanol) was subdivided in 21 ranges, with n-butanol being in the 8th range. The rear boundary of each range was equal to the front boundary's retention time plus 0.02 plus 12 percent of the front boundary's retention time. The intensity of the range was equal to the sum of areas of all peaks having apexes within the range; the actual intensity was transformed logarithmically, with conventional linear increments, to form the intensity vector for the discriminant analysis.

At that stage, three statistical experiments were conducted. In one, discriminant functions were formed using 27 human chemical signatures and 5 chemical signatures of air from a cafeteria kitchen area taken at different times. The separation in terms of two

discriminant functions, X and Y, after computer use of 6 out of 21 variables, is shown in the top part of Figure 15. The derived discriminant functions were used to compute X and Y for additional 24 human chemical signatures. The values obtained were plotted over the repeat plot of the top portion of Figure 15, in the bottom part of Figure 15. Reasonably successful classification of additional signatures was observed with 15 out of 24 falling into the "range circle" somewhat arbitrarily drawn, and several more closer to the center of gravity for the human patterns than to the cooking vapor pattern center.

The second experiment is represented in Figure 16. Here 37 chemical signatures were classified in three groups: 20 Caucasian males, 12 Indian males (students from India, mostly vegetarians) and again 5 cooking vapor samples, using up to 14 variables.

The third experiment consisted of classifying chemical signatures in two groups: 16 females and 35 males. The classification after using 7 variables is shown in Figure 17. This experiment is significant since here only the chemical signatures obtained in the glass cell apparatus (cf. Section 3.1) were used, and the possible question on discriminant features introduced by sampling humans in the cell versus sampling environments in air at large does not arise.

At this stage of the work, normalization of the retention times in terms of Kovats Indexes was introduced (cf. Section 3.6.5) and it appeared desirable to subdivide the chromatogram into Kovats Index increments for the purpose of the stepwise discriminant analysis. This index is a logarithmic function of the retention times, hence equal index intervals approximate equal retention time percentage increments.

One set of the statistical tests was conducted using arbitrary division of the chromatogram into ranges 50 Kovats Index units wide. Thus, 22 ranges were set up: one with K.I. below 600, then 20 ranges each 50 units wide, and the last range with K.I. above 1600. This converts each chromatogram into a set of 22 variables; the intensity

of each variable is equal to the sum of logarithmic transforms of all peak areas with the apexes in the given range. Two discriminant analyses were conducted. One used three groups: 39 males, 26 females, and 10 environments. The other used human signatures, obtained in the glass cell only, and into 3 groups: 25 Caucasian males, 12 Indian males, and 26 Caucasian females, 63 humans in total. Tables XIII and XIV illustrate the success of classifications, in terms of percent of patterns properly assigned to the correct group. Discriminant functions based on 13 variables produced more than 80 percent of correct assignments for the test containing 63 human chemical signatures. Most of the classification was achieved at taking 5 variables only. It can be noted that sometimes upon inclusion of further variables (cf. variables Nos. 3 and 4, female column Table XIII) the percentage of correct assignments can decrease. This effect results from compromises when optimization of group separations leads to a larger deviation from the group center point for an occasional case.

All these experiments conducted by mechanical separation of gas chromatograms into range variables indicated that discriminating features can be found and reasonably successful functions formed after using only a fraction of the variables, but that 5 to 15 variables may be needed to sufficiently define the chemical signature source if the identification is based on vapor gas chromatograms consisting of 30 to 50 peaks.

At this juncture it was decided to explore if a more selective choice of variables (gas chromatographic ranges) can result in better and more valid classifications, or help to pinpoint, for the future gas chromatographic and mass spectrographic identification those narrower ranges where significant discriminating features reside.

4.2.3 Stepwise Discriminant Analysis With Selectivity Chosen Variables

In this part of the statistical experiments, attempts were made to use several rationales in selecting gas chromatographic ranges to serve as variables in the input to the stepwise discriminant analysis program, instead of mechanically dividing the entire gas chromatographic range into a set of adjoining ranges, as was done in the work described in Section 4.2.2.

Histogram Procedure. Here data on the positions of gas chromatographic peaks expressed in Kovats Index units were pooled for all 65 patterns of humans together, or for males and females separately and arranged in the order of increasing Kovats Index. Wherever peaks occurred frequently in the human chemical signatures, the number of peaks, e.g., per 10 Kovats Index units was higher than elsewhere. Figure 18 represents the histograms of the frequency of occurrence of peaks in Caucasian males and females along the Kovats Index scale.

These histograms can be considered as generalized gas chromatographic patterns, with the peaks indicating those ranges where peaks of components most commonly occur; the peak areas of the original gas chromatograms are not indicated by this plot, except that only those original peaks were considered (about 2/3 of all peaks) which were larger than 40 percent of the average area of peaks in the corresponding pattern. There are striking similarities between the occurrence of peaks with the Kovats Index values in the two generalized patterns, but also distinct differences; for instance, substance with K.I. = 1030 (asterisk) much more frequently appears at larger relative concentration in effluents from females than from males. The peak areas are not considered in the histograms.

More histograms were prepared using an available computer program in which the pooled and ordered Kovats Index array is scanned, taking each peak in turn and calculating the Kovats

Index interval between two peaks, the first of which is, e.g., 10 peaks before 1 and the second is 10 peaks after 1. Wherever this interval is smaller, the peaks occur more frequently. An example of a part of such printout, which is in essence reciprocal to Figure 18, is shown in Figure 19. The points of minima represent those locations on the Kovats Index scale where peaks occurred more frequently.

Selection of Variables. By inspection of a histogram for the entire gas chromatographic range of 65 pooled human chemical signatures, 37 locations were found where the peaks occurred most frequently, that is the minima in the histogram such as shown in Figure 19 were most pronounced. The variables were set to center on the minima and to have a range of ± 10 Kovats Index units around the selected minima. All peaks with apexes in the K.I. range were considered to be fully within the range. Thus, the chromatograms were each converted to 37 variables; cf. Section 3.8 for the intensity transformation procedure used to assign the intensities to the variables. If the center points of two adjoining ranges were closer than 20 K.I. units, the boundary between the ranges was set at the middle point between the centers. This selection left 15 to 20 possible high frequency-of-occurrence ranges not included.

The 37 ranges, called P-ranges (peak-containing) were adjacent at some locations, but separated by ranges in which peaks in the chemical signatures of humans were less common. Table XV lists all types of ranges, consequently numbered, with indication of the range widths and the type of range (P or non-P). The statistical discriminant analyses were conducted using various choices of variable sets. Thus, all 37 P ranges, or 10 of P-ranges, or the non-P ranges were tried; finally, 34 variables were selected, shown in the last column of Table XV, which included 18 P-ranges shown to be most discriminating between human and environment chemical signatures and 16 non-P ranges, also shown.

among the non-P ranges, to be most discriminating. The consideration of non-P ranges was made upon the suggestion of personnel of the Ballistics Research Laboratory who pointed out that the pattern discrimination should be aided by considering, in addition to the presence of certain features, also the lack of other features.

Stepwise Discriminant Analyses. Selected typical experiments are summarized here.

Experiment 1. Discrimination of 65 Human from 10 Environment Chemical Signatures Using P-Ranges Only. Table XVI summarizes the efficiency of classification.

In run A, all 37 variables were submitted; in run B, those ten variables ranges were selected from the 37 ranges where the peaks most frequently occurred in human chemical signatures. It is seen that in test B, the computer needed to use only five ranges as variables to separate 91 percent of the patterns from humans into one coherent group. In test A, 94 percent coherence was accomplished after the use of 11 ranges out of 37, with no improvement beyond that. After the extraction of 17 variables out of 37, the computer reported that no information that would be statistically useful for group separation was detectable in the remaining 20 variables. The degree of coherence in classification reached by this technique of the selection of variables was considerably better than that obtained by a division of the patterns into ordered ranges without regard for the inner logic of the patterns. On the basis of data presented in Table XVI, component identification can be centered on the most significant ranges. It must be noted that the important ranges can include several peaks so that a decision must be reached, by the use of higher resolution columns, as to which are the important peaks within the important range.

Experiment 2. Discriminant Analysis of Human Versus Environment Chemical Signatures Using P and non-P Ranges. Here two runs were made. In Run C, 36 human chemical signatures were selected at random from 65 available signatures, and all 10 environment chemical signatures; the discriminant analysis was conducted using all P-ranges only (marked P in the "Range Width" column of Table XV). In Run D, the discriminant analysis was conducted using all other, non-P-ranges only. Figure 20 compares the relative efficiency of both selections.

Classifications based on either set of variables were eventually comparably successful. The classification based on those Kovats Index ranges in which peaks were frequently observed in samples from humans was more efficient when a smaller number of variables was utilized.

Experiment 3. Derivation of Discriminant Function Using Part of Data and Test of the Function Using the Rest of Data. Here a new selection of the variables (34 in total) was made. It included the most statistically significant variables both from the Kovats Index ranges in which peaks frequently were observed in the vapors from humans (P-ranges) and the intervals between these ranges. The selected variables are marked by crosses in Table XV with the P-ranges also marked with P. Two runs were selected for the report.

In Run E, 36 human chemical signatures, randomly selected from a total of 65 available, and all 10 environment chemical signatures were used to derive the discriminant function. Then the same discriminant function was applied to classify the remaining 29 human chemical signatures. Figure 21 shows that only 4 out of 29 or 14 percent were misclassified if the discriminant function based on 15 variables (computer-selected from 34) was used. The variables were, cf. Table XV, Nos. 18, 30, 35, 37, 44, 47, 49, 54, 55, 56, 57, 59, 61, 63, and 65.

If the discriminant function derivation was stopped at a

fewer than 15 variables, class separations were somewhat poorer:

<u>Variables Included</u>	<u>Percent Correctly Classified in Testing The Discriminant Function</u>
2	71
5	74
15	86

In Run F, a three-way classification was tried. The discriminant functions X and Y were derived for chemical signatures of 26 females of several races, 22 males of several races, and 10 environment samples, or 58 patterns in total; 15 additional male chemical signatures were kept if the additional males were correctly classified by the derived function.

The male group used for derivation consisted of 14 Caucasian and 8 Indian males; the male group withheld for testing contained an additional 11 Caucasian, 2 Negro, and 4 Indian males. The progress of classification during the function derivation is shown in Figure 22. After use of 20 variables out of 34 variables available to the computer, the statistical noise level was reached, with 4 females still not correctly classified. Figure 23 shows the classification field in terms of two discriminant functions X and Y. The hollow triangles represent the male chemical signatures used in derivation. The filled triangles represent the male chemical signatures used to test the functions. Although an intrusion into the environment ("Airs") chemical signatures region occurs, the test points all are far away from the region of the field populated by the female chemical signatures. This experiment again indicates that classifiable features exist in the chemical signatures of males and females.

Other Statistical Experiments. A number of other class separations was tried, e.g., Caucasian males versus Caucasian females versus Indian males, and some four- and five-class separations. None of these was 100 percent successful, but varying lower percentages of success were achieved.

The pattern stepwise discriminant analysis led to the conclusion that under conditions where the chemical signatures are expressed in terms of up to 30 variables, reasonable classifications by the classes of sources (sex, race, humans versus environments) becomes possible with use of 2 to 20 variables. The required number depends on the similarity between the sources; in the case of humans versus environments as few as two to five variables suffice to effect 80 percent levels of success. The derived discriminant functions held reasonably well under tests with additional chemical signature patterns.

The results also indicated those Kovats Index ranges which contain components most useful for the source discrimination and therefore most suitable targets for further chemical identification.

4.3. Three Column Data

The three-column gas chromatographic device (Section 3.6.4) produces readout which can be analyzed in a two-fold manner.

Three Point Match. For the compounds for which the three time points of exit (from the 200 ft Carbowax 20M column and subsequently the short and the long Apiezon L columns) have been established, an inspection of the three gas chromatographic recording tracks permits judging if these compounds are present in the sample of chemical signature. As in any judgement based on gas chromatographic retention times in several stationary phases, there are two types of errors possible: (1) the location of the peak of a known compound can be shifted because of other materials present, so that a slight non match does not rule out the presence of the compound and (2) other compounds can produce peaks in closely similar locations so that a slightly non-matching point can be erroneously taken to represent the particular compound. In the case of a three-column device, with a continuous flow of the components through the system, additional erroneous judgements

are possible. "Coincidence ghosts" can arise if a three-point match, or near-match, results from simultaneous presence of several components; one component may leave the Carbowax 20M column at the same time as the known compound, while other components which left this column at different times can leave the Apiezon L columns at the time points expected for the known compound. These errors can be reduced if the known compound can be added to a duplicate sample and the sample reanalyzed; if the peak in all three points shows no indication of broadening or splitting, the evidence that the known is present in the unknown is very considerably strengthened, while if the peak broadens or splits, errors of coincidence or presence of additional gas chromatographically similar components are likely.

In Kovats Index system, approximate prediction of the gas chromatographic positions of compounds is possible from rules operative in this system, (cf. Section 3.6.5).

Table XVII lists some three-point matches obtained by comparisons of chemical signatures of humans with the three-point locations of known compounds run through the three-column device independently.

The three-point process, if it is to be utilized either for the components pre-identification or as a part of pattern analysis, must be studied to establish: (1) the experimental reproducibility of the positions of the same compounds in the context of various mixtures, (2) the probability of occurrence of other compounds that can produce, each completely by itself, similar sets of three time points, and (3) the probability of coincidental production of a similar three-point set, by two or three different components.

Computerized Indexing. Here, peaks are matched without reference to known compound points, except that an n-alkane calibration scale is used to convert the readings to Kovats Indexes. A program GA3COL, was written for IBM 360 computer to process the three-column gas chromatographic data and print out the most

probable sets of Kovats Indexes in Carbowax 20M and Apiezon L for the components indicated by peaks.

Each peak emerging from Carbowax 20M column enters both Apiezon L columns simultaneously. The ratio of the corrected retention times on the long: short Apiezon L columns is approximately constant. After calculating this ratio for each Carbowax 20M peak as the possible origin and for the various combinations of Apiezon L peaks within the gas chromatographically feasible retention time range and applying the ratio \pm tolerance criterion, the computer prints out those peak sets that meet the criterion, with the Kovats Index data calculated on the basis of calibration with n-alkanes.

The ratio was explored using known compounds. Its average was 2.05. The critical point is the tolerance allowed. If the tolerance is too small, some components actually present in the sample can exhibit ratio which because of normal experimental variations would be outside the limits and would not appear in the tabulation. If the tolerance is too broad the frequency of mathematical coincidences meeting the limits will begin to increase. On the basis of preliminary experiments tolerance of ± 0.10 was judged reasonable. The computer program can be run with any selected tolerance.

Figure 24 illustrates the data obtained by the computerized analysis of the three-column gas chromatograms of vapors collected from the same subject simultaneously. The figure combines two analyses, at 90° and 150°C. It indicates the utter complexity of the composition and the possible presence of groups of compounds indicated in the upper right corner.

Later work with the two-column system (Sections 3.6.3 and 4.3) confirmed the complexity of peaks eluted from the 200-ft Carbowax 20M column. Since the two-column data were easier to interpret, and a more detailed study was desired in the gas chromatographic ranges that were significant in pattern

classification (cf. Section 4.2.2 and 4.2.3), the work with the three-column system was set aside in favor of that with the two-column system. The limited experience with the three-column system operated isothermally led to the following observations: (1) the system, in principle, can be useful for a rapid overview of new patterns (2) since small errors in short retention times strongly influence the ratios of the retention times, the matching of peaks at the beginning of the gas chromatograms, and for polar components with short retention in Apiezon L throughout the chromatogram, is less certain, so that components actually present can be missed if tolerances there are set too narrow, (3) towards the end of gas chromatograms, resolution increases to the extent that several peak combinations can reasonably meet the matching criterion.

It is evident that the ease of interpretation of the three-column data significantly depends on the number of components present in the sample and on the resolution of columns used. Human chemical signatures exhibit complexity that limits a straightforward interpretation of isothermal analyses.

4.4 Two-Column Data

The data on tentative identification of selected components of human chemical signature acquired by means of two-column apparatus are displayed in Table XVIII. The data were derived from the plot in Figure 25. The Carbowax 20M peaks examined by this technique were arranged in order of increasing Kovats Retention Indexes and numbered accordingly. The selection of peaks to be examined was based on Kovats Index Ranges isolated by the computer program. The ranges comprised peaks with maximum occurrence frequency in chemical signatures of all subjects previously studied. The computer-isolated ranges together with the number of peaks observed in the ranges are given in the second column of Table XVIII. Except for Carbowax 20M peaks 1 and 3, all peaks examined fall within the selected ranges:

peak 1 preceeds the range by only 7 Kovats Index units and peak 3 by 12 units.

Since the pattern analysis was based on gas chromatographic data obtained with packed partition column while Table XVIII is based on data from the 200-ft open tubular column with much higher resolution, an exact correspondence between peak positions in the two sets of data is not to be expected.

The twenty-five parent peaks selected in elution from the first (200-ft Carbowax 20M) column resolved, as Table XVIII indicates, into over 150 components on re-analysis in the 50-ft Apiezon L column. The number of components per peak ranged from 3 to 10. Contribution of these components to the parent peak varied over a broad range, but usually one to three daughter components could account for the most of the parent peak area. Thus, the 25 parent peaks resolved in the second (Apiezon L) column, into 36 major components and approximately 120 minor components. The major components are marked by asterisks. Some parent peaks, e.g., one with K.I.=1540, resolved into several components none of which relatively large with respect to the others, so that none of the daughter peaks could be asterisked.

The two Kovats index values for each peak partially characterize the type of the compound. The most convenient identification guide line is through calculation of a difference or dispersion parameter:

$$(K.I. \text{ in Carbowax 20M}) - (K.I. \text{ in Apiezon L})$$

This parameter describes the gas chromatographic "polarity" with respect to Carbowax 20M stationary phase. The parameter is zero, by definition, for n-alkanes; can be a small number for branched alkanes; and increases by approximately 70 units by presence of a double bond in the hydrocarbon. The difference parameter is given in the corresponding five columns of Table XVIII, where it is classified into five ranges.

The first range, 0 to 100, contains alkanes and alkenes. The second range, 100 to 250, may contain dialkenes, ethers, acetals.

substances with heteroatoms in saturated or one-double-bond ring. The third range 250 to 380, is populated by alkanals (aldehydes), ketones, esters. It also can contain benzene and its alkyl derivatives; however, benzene has a Kovats index pair (in Carbowax 20M/Apizeton L) 960/680; toluene 1060/796; o-xylene, 1213/926; m-xylene, 1173/901; p-xylene, 1166/899; and ethylbenzene 1152/884; none of these is represented within error limits of order of ± 10 K.I., in the table and hence most likely are not present among the over 150 compounds shown. Amines also can be present in this range. The fourth range, 380-520, contains alkanols, lower alkenals, and alkadienals. The fifth range, above the dispersion parameter of 520, can contain substances with two polar groups on the molecule, or with several double bonds on alkanol molecules. The lowest member of glycol series, ethyleneglycol, has the index pair of 1580/660, and is not present in Table XVIII.

The indicated range boundaries are only approximate, especially at the lower end of K.I. values. This can be illustrated by the following examples. The n-alkanals are located at discrete K.I. pair values separated by approximately 100 unit steps between the successive members of the series: n-pentanal 985/685, n-hexanal 1078/783, n-heptanal 1188/867 (assumed, in Table, 1220/865), with the dispersion parameter of 300, 295, and 321, respectively. If the polar functional group is partially shielded by non-polar side chains, the molecule behaves in less polar manner; thus

n-butanal	= 326
2-methyl butanal	291
n-hexanal	295
2-ethylhexanal	271

Double bonds, on the other hand, increase the gas chromatographic polarity, cf

2-ethyl hexanal	271
2-ethyl-2-hexenal	341
n-butanal	326
n-butenal (crotonaldehyde)	433
2,4-hexadienal	509

The index pairs, of course, also are located at a point different from the corresponding n-alkanal points. Similar rules apply to ketones and esters:

Butyl Butyrate	$\sigma = 287$
Isobutyl Butyrate (polar locus shielded)	233
Ethyl butyrate	285
Vinyl butyrate	304
Propyl propionate	286
Isopropyl propionate	270
Propyl acrylate	328
Allyl acrylate	392

Similarly, for n-alkanols the dispersion parameter is in the range of 480-510 (except for the lowest members of the series), decreases if side alkyl groups obstruct the hydroxyl group, and increases if double bonds are introduced.

Thus, although the grouping by ranges in Table XVIII is not ideal, it gives insight into the nature of the components. With the knowledge of the Kovats index in Apiezon L and of the approximate gas chromatographic polarity, estimates of the boiling points of the components are also possible and can be considered to be within $\pm 10^\circ\text{C}$ of the actual value. The estimated boiling points of the components are also tabulated in Table XVIII.

An absolutely positive identification of the specific components from the two gas chromatographic parameters only is not an exact procedure; it merely eliminates many unlikely choices, narrowing the selection to a few possible candidates. Pending the mass spectrographic identification, a closer chemical characterization of the over 150 components was not conducted. Some points fitted reasonably well certain compounds, however; these are indicated in parentheses as the most likely choices in the last column.

A survey of Table XVIII invites several interesting generalizations.

First, there is an unexpectedly frequent presence of alkanes and alkenes, with eight to fourteen carbon atoms, and with C_{12} alkane either not represented or present at low concentrations.

This can be either a physiological effect, or an effect that characterizes urban culture with vehicular exhausts containing unburned hydrocarbons present in air and picked up by human subjects. There are 35 components of this type; 9 of these are among the major 36 daughter-components. Some negative dispersion parameters that show up in the table are inherent in the peak-reinjection procedure where the injection into the second column is considered to occur at the middle of the finite duration injection interval, (e.g., 30 sec) although actually some components are present in the leading edge, others in the trailing edge of the parent peak. An alkane contained in the trailing edge of the injected parent peak will result in a small negative dispersion parameter.

The carbonyl compounds (including esters), with amines also possible, occur in the third dispersion index column, with possible additional cross over into higher and lower ranges. Here, 62 components occur, among these 19 are major components of their respective parent peaks.

The second dispersion index column contains 29 components, but only 2 of these appear to be major components of the respective parent peaks.

The fourth dispersion index column (alkanals, alkadienals) contain 22 components; 5 of these are major.

The fifth group contains 10 components, one of these is a major contributor to the corresponding parent peak. These are multifunctional substances or polyenes with polar functional groups, and their presence may deserve further exploration since they begin to appear in the higher boiling point ranges, repeatedly indicated to be important in discriminant functions. Also, compounds with lesser volatility can be recovered from air with higher efficiencies and perhaps more suitable in detection devices.

To summarize the inspection of Table XVIII, relatively high frequency of hydrocarbons, and the dominance of carbonyl compounds (including perhaps esters) among the major components, and

presence of multifunctional compounds, or carbonyl compounds with more than one double bond appear to be the characteristic features of the human chemical signature studied by the high-resolution two-column technique. Approximate group characterization of the components is summarized as follows:

	Major Components of Parent Peaks	Minor Components of Parent Peaks	Total
Hydrocarbons	9	26	35
Acetals, Ethers, Heterocyclics	2	27	29
Carbonyls, Esters, Amines	19	43	62
Alcohols, Diene Carbonyls	5	17	22
Diene Alcohols, Polyfunctionals	1	9	10
Total	36	122	158

*A few duplications, for peaks close in Kovats Index values on Carbowax 20M column, can be possible.

An inspection of Table XVIII also indicates that from the 25 parent peaks studied, the peaks areas reasonably well represent primarily the content of one single daughter component in 12 cases (one major daughter peak found in each), represent primarily the content of two daughter components in 11 cases, of three daughter components in 3 cases, and none in one case. In the latter, the parent peak consisted of several daughter components none of which excelled in magnitude. These observations should assist in considering the merits of single-column devices vs. two-column devices for pattern classification, at the resolution levels obtainable with 200-ft columns. The presence and the changes in the concentrations of the minor daughter components can, however, influence the position of the apex of the parent peak in the primary gas chromatogram and under certain conditions influence the pattern classification data, by shifting the peak assignment across a range boundary into the range of neighboring variables.

Therefore, the significance of the minor peak components in pattern recognition cannot be ignored.

Aside from the above 25 peaks eluted from the first 200-ft Carbowax 20M column and re-analyzed in the 50-ft Apiezon L column, the gas chromatogram from the first column contained many additional peaks. Data obtained from vapors of the same subject, a Caucasian female, and analyzed at 90°, 120°, and 150°C are listed in Appendix II. There is reasonable similarity between two analyses at 150° (A and B) on two subsequent days from the same subject. A summary of the number of peaks observed in vapors is as follows:

Kovats Index Range	Analysis Temperature, °C				Maximum Number in Respective Range
	90	120	150 (A)	150 (B)	
Below 300	10	2	3	3	10
801-1000	11	11	7	4	11
1001-1200	25	16	6	5	25
1201-1400	11	27	12	13	27
1401-1600		9	20	25	25
1601-1800		2	25	21	25
			7	3	7
TOTAL	57	76	80	74	130

Thus, prior to the subsequent split in the Apiezon L column, a 200-ft Carbowax 20M column, used isothermally at three different temperatures, indicating the presence of approximately 130 peaks.

4.5 Mass Spectrographic Identification of Human Signature Components

The initial results showed that the sizes of the peaks of both human signatures studied fell below the sensitivity of the mass spectrograph.

The experiments on the mixture of n-butanol and heptanal showed that both compounds were positively identified each time their respective gas chromatographic peak sizes matched those of the major and medium-size components of human signature.

The standard gas chromatographic analysis of the human signature collected on the large fluidized bed showed a significant increase in the sample magnitude. The size of the sample found was at least ten-fold that of the sample usually collected on the standard bed. Also, the peak distribution pattern was similar to the pattern previously obtained from the same subject under routine collection conditions.

Mass spectrographic analysis of human chemical signature collected on the large bed and fed directly from the open tubular column produced patterns which in most cases were too complex for immediate interpretation. One analysis indicated the presence of a compound (naphthalene) probably transferred to human from moth-producing materials. The peak of this compound was usually well resolved as observed in the respective gas chromatographic analyses of the sample.

The direct mass spectrographic analysis of effluent carrying human samples collected on standard size beds produced patterns which in all cases studied were too low above the instrument background for positive identification.

The difficulties encountered in the mass spectrographic identification of human signature components result mainly from the lack of sample resolution rather than its size. This is supported by the positive identification of the naphthelene (since its peak was well resolved), by the results of experiments on 1-butanol and heptanal and by recent frequent observations made

during the analysis of human signatures on the two-column apparatus. The results of these experiments indicated that most Carbowax 20M peaks were multi-component peaks.

5.0 PROGNOSIS FOR CHEMICAL SIGNATURE DISCRIMINATION DEVICES

The work conducted indicated that the complex chemical signatures of humans and environments contain features which permit classification by source type using relatively straightforward statistical discriminant techniques. The number of features that must be taken in account and their distribution within the gas chromatographic spectrum depends on the type of sources that must be discriminated. Although it is possible that further research may uncover some specific substances or a few substances that characterize e.g., humans versus environments, it is questionable if signature detection and identification devices should be based entirely on such features. A device that can be based on sufficient redundancy of features is less likely to suffer from interferences. From the findings thus far, it appears that 7 to 15 complex features may be necessary to reasonably characterize the source. The two-column data also indicate that these features consist of several chemical components some of which probably represent useful features while others act only as chemical noise.

Two questions can be posed: (1) which processes are best suited for signature identification and (2) can a sufficiently rapid device be envisioned?

Since the useful features are represented by various molecular species, it would be desirable to use vapor separation methods that permit retention of the molecular identities rather than other processes (such as mass spectrometry on non-separated mixtures) which splitter the molecules. Such methods are the basis of gas chromatography. Separation by interaction with a polar medium, as used in the present work, permits locating the discriminating features. It appears that a useful discriminant system can be based on the principle of preseparation in polar columns and inspection of the discriminating regions in the second column or columns, cf. 4.4. Vertical cuts through e.g., Carbowax 20M versus Apiezon L field plot in the regions of interest should produce

enough information for identification of source types.

The second question, on the order of speed that may be achieved by such devices, must be answered in two steps: can a preconcentration of organic vapors from air be conducted fast enough, and can the subsequent task of identification of the features be conducted fast enough?

Concentration. Although higher sensitivities to all or to some substances can be achieved with some gas chromatographic detectors especially if helped by very careful work and adjustments, nanogram (10^{-9} g) amounts of individual substances of all types can be relatively easily detected by devices widely used in routine gas chromatographic practice. For a typical substance with a molecular weight of 100, this corresponds to 6×10^{12} molecules. Assuming the desired detectability level at 10^9 molecules/cm³ of air and the maximum gas-sample size into the device's processing channels at 1 cm³, a concentration factor of 6000 is needed to bring the lowest concentrations into the detector's performance range. Solubility of vapors of organic materials in gas chromatographic stationary phases decreases by a factor of such magnitude when temperature is changed from ambient to above 200 to 250 °C. Hence, equilibrium absorption from air at ambient temperatures and desorption at above 200 °C, in principle, permits one-step concentration of the dilute chemical signal to the level needed for a routine detection by usual gas chromatographic detectors.

There is no limitation, in principle, why such a process cannot be conducted with speeds of order of 1 sec. A thermodynamic requirement and a kinetic requirement must be met.

$$(k) (n_a) (v) > 6 \times 10^{12} \quad (I)$$

$$\frac{x^2}{2D} > 1 \quad (II)$$

where

k is the dimensionless partition coefficient for the vapor in the stationary-phase/air system, equivalent to the factor by which the concentration of the vapor molecules in the solution in the phase is larger than their concentration in air when equilibrium is reached

n_a is the concentration, in molecules/cm³, of the vapor in air

v is the volume, in cm³ of the stationary phase, in the concentrating device

x is the thickness of the absorbing film of the stationary phase in the device, cm

D is the diffusion constant of the vapor molecules in the stationary phase, cm² sec⁻¹

Partition coefficients at ambient temperature for vapors of substances with ambient saturated vapor pressures of several millimeters or lower in appropriate stationary phases easily reach into the range of 10^3 or higher. Therefore, the first requirement satisfying the sensitivity of the detectors can be met, for $n_a = 10^9$, with a few cubic centimeters of a suitable stationary phase. The second requirement, from the diffusion laws, must be met to perform absorption equilibration in a time span of order of 1 sec. With a typical $D = 10^{-8}$, the permissible film thickness is of order of 10^{-4} cm, well within technologically feasible limits.

Resolution. The complete resolution of the chemical signal captured from air would consist of a full inventory of the molecular species present in air at the time of inspection by the analog. As in any process, compromise must be accepted between resolution and time. Resolution is possible at two stages of processing -- in sending the vapor mixture that constitutes the signal with assistance of a carrier gas through a gas chromatographic partition tube, and in using species-selective detectors on the signals resolved in the first stage. Most detectors and their electronic circuitry have very fast response, and their specificity cannot be improved by simply allowing more time. Therefore, signal processing time and resolution depend

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primarily on partition-tube performance.

The resolution that can be produced per second can be estimated from the following equation*:

$$\frac{R_{23}}{t} = (r_{23} - 1) \frac{k}{(1+k)^2} \frac{\sqrt{N}}{t_m}$$

where

R_{23} is the resolution of the vapors of substances 2 and 3 ($R_{23} = 1$ indicates very distinct resolution with a negligible overlap of signals)

r_{23} is the ratio of partition coefficients of the vapors 2 and 3 in the stationary phase at resolution temperature

t is the time in seconds

k is the retardation ratio in the partitioning tube and depends on the volume of gas space, the stationary phase in the tube, and the partition coefficient of the vapor in the stationary phase

N is the equivalent plate number of the partitioning tube

t_m is the gas holdup time (volumetric flow rate of carrier gas through the tube divided by gas phase volume)

We now use this equation to estimate what ratio, r_{23} , in partition coefficients of 2 and 3 can be ascertained in 1 sec ($R_{23}/t = 1$) in a typical short partition tube. For capillary tubes, k is of order of 0.2 to 20, and a 10-cm-long tube can typically be equivalent to $N=100$. Selecting $k=1$, and $t_m=0.1t$, $r_{23}=1.04$. A difference of 4% in the partition coefficients of 2 and 3 should suffice to tell these components apart within 1 sec. Other estimates** show that a "100-plate" partition tube can permit recovery of substance 2 with less than 5% impurity of the substance 3 and, in reverse, if both are present in equal amounts

*Loyd, R.J., Ayers, B.O., and Karasek, F.W., Anal. Chem., 32, p. 43, 698 (1960).

**Littlewood, A.B., Gas Chromatography, p. 136, Acad. Press, New York, 1962.

in the original mixture and their partition coefficients differ by 40% or more.

In the case of very complex vapor mixtures, many compounds are present, and the above resolution is insufficient for a complete inventory. There is a continuous overlapping of the substances with small differences in the retention times. In usual cases, low-resolution processing then results in a poorly differentiated effluent from the partition tube with peaks of noticeable size concentrations considerably in excess of the total concentration of other poorly differentiated substances. Any portion of the effluent from the above "100-plate" partition column, taken during the time interval of $t \pm 0.4t$, contains over 95% of the component or components with the retention time t , and a lesser fraction of components with retention times within the $t \pm 0.4t$ boundary.

In the notation of Kovats retention index (cf. Section 3.6.5), the retention times are referred to the scale of n-alkanes with a logarithmic transformation to produce intervals of 100 Kovats Index units between each of two consecutive members of the homologous series of n-alkanes. Two compounds with retention times of t and $t + 0.4t$ produce an index difference of 50 K.I. units. The interval between C_2 (200 K.I.) and C_{20} (2000 K.I.) alkanes carries 36 intervals of 50 K.I. units each. Thus, in principle, a parallel multichannel unit with separate non-polar phase channels operated at different processing regimes (temperatures, flow rates, and times) can split the Kovats Index continuum corresponding to compounds with boiling points up to 400°C into 30 to 40 cells. Each cell is populated by molecular species with relatively similar partition coefficients in the gas/non-polar phase system. The detector response at the exit of each low-resolution channel is a complex function of the population distribution within the cell with frequency maxima at those time points at which some more-than-usually abundant molecular species elute.

Various discriminators can then be applied to the population of each of the cells. Two types of discriminators can be derived from gas chromatographic practice. One is a polar or otherwise specific stationary phase in a low-resolution column with its characteristics being the shift of the elution positions with respect to the n-alkane position. The other is the sensitivity of the detector to some atomic species, e.g., a sulfur-sensitive detector indicates the distribution of sulfur compounds among the cells and within each cell. The species-sensitive detector can apply to the initial multichannel signals or to the signals first distorted by the second-stage discriminator columns. Since the dispersion shifts easily exceed 50 K.I. units, a low-resolution discriminator is sufficient to produce a rather informative dispersed signal. The shifts are characteristic of functional groups in the molecule and of the steric clustering of non-polar parts of the molecule around the functional group.

Thus, there is no fundamental difficulty in development of processes that can characterize chemical signatures of humans within a few seconds to a degree sufficient for classification by sources. The actual choice of the processing steps requires however a massive experimental and theoretical effort.

6.0 SUMMARY AND CONCLUSIONS

Within the context of the present work, chemical signatures are defined as compositional characteristics of organic vapors emanated into air by various sources. The objective of the conducted work was to establish if features exist in the chemical signatures of humans that can be used to distinguish humans from environments and to distinguish among humans. A further objective was to characterize those features as far as possible within the limits of the effort and presently available technology. An all-glass Teflon cell was available from the previous project (DA-11-022-AMC-1775(X)). This cell permitted isolating humans temporarily, from the surrounding air and maintain them in a flowing, highly purified air stream so that better samples, not confused by a continuous arrival of contaminants from air, could be collected.

It was soon discovered that many of the usual sample collection procedures, such as: collection in vessels, cryogenic cooling of air, or adsorption on carbon, were unsuitable for the chemical signature acquisition when the chemical signatures consisted of a large variety of organic materials available in air at concentrations as low as parts per billion.

Consequently, considerable effort was directed toward the development of improved techniques for the acquisition and processing of samples of chemical signatures from humans and various environments. The essential features of this development involved new systems for sample collection, transfer, and injection into the gas chromatograph. The sample acquisition was based on the concept of collection through equilibration. The collectors were special fluidized beds of Teflon powder coated with a thin film of Apiezon L stationary phase. The fluidized bed configuration of the collector produced the best possible contact of samples with air. The Apiezon L permitted later recovery of the dissolved sample with minimum artifacts from decomposition of the trapping phase itself. In a typical

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collection of a sample from a human, the bed material was exposed to air carrying the vapors at the flow rate of 0.75 liters per sec. The environment chemical signatures were collected by pulling the air through the sampler by a small blower. Since the collection involved equilibration, the actual airflow rate through the sampler did not have to be known and was not critical as long as the powder was kept in the fluidized state.

A special calibration procedure was devised to correlate the concentration of a known sample component in air directly to the size of the peak on the gas chromatogram, to check the collection efficiency and to establish the time required for attainment of equilibrium concentrations for various types of compounds.

The recovery of the collected sample from the trapping film and its injection into the gas chromatograph for analysis was effected by a special sample transfer and injection system. The sample transfer system was designed to allow the transfer of the vapors from the bed maintained at 80°C into a special stainless steel thin wall needle which was kept at liquid nitrogen temperature. The transfer was effected with cryogenically purified gas. The sample injection system was constructed to deliver the sample from the injector needle to the gas chromatographic column in a timed, automatic and reproducible manner.

The sample acquisition step was found to require 45 min to produce reproducible and equilibrated samples. The typical duration of the sample transfer step was 6 min. During this period 70 to 80 percent of the vapor sample was found in the injector needle. Higher efficiency could be achieved with a longer transfer time but at the expense of increased analytical time. The sample injection step resulted in reproducible and practically quantitative injections with 95 to 98 percent of the sample transferred from the needle and introduced into the injection port of the gas chromatograph.

The standardized techniques developed in the initial phase

of this work were used to obtain 65 gas chromatographic patterns of human signatures and 10 patterns of various environments. The human signatures were collected from healthy subjects of both sexes and several races. The races included American Caucasian males and females, Indian males (born in India), a few American Negroes, and a Mexican and a Malayan subject.

The first series of chemical signatures were analyzed at 125°C using a 10-ft, 1/8-in O.D. packed bed columns with a Carbowax 20M stationary phase. Later work, conducted on a smaller variety of samples from humans, utilized 50 and 200-ft long support-coated open tubular columns with a Carbowax 20M coating. These provided much higher resolution and were used isothermally at several temperature levels (90°, 125°, and 150°C). In still later work, a specially modified two-column gas chromatograph was used in which the sample was first analyzed in a 200-ft support-coated open tubular Carbowax 20M column and the gas chromatographic peaks found previously to occur most frequently in the human chemical signatures were further analyzed in a 50-ft support-coated open tubular Apiezon L column. This procedure allowed the resolution of peaks into further components and a subsequent estimation of their gas chromatographic polarities. A three-column gas chromatograph was also used, combined with a computer-analysis of data, to characterize the components of the chemical signatures in similar terms.

The great complexity of the chemical signatures was revealed by the experimental survey of the samples from 65 humans, and 10 environments in analysis in the 10-ft packed columns. At this stage, the data were studied by several computerized statistical analysis techniques, to discover if: (1) valid discriminant functions can be extracted permitting classification of the signatures by the types of their sources, (2) how many gas chromatographic features need to be used to obtain classifications, and (3) indicate those features which are most significant in forming discriminant functions and therefore deserve priority in

their chemical identification.

Attempts at mass spectrographic or other identification of components at various degrees of gas chromatographic resolution were also made. These indicated that the highest degrees of resolution in a two-column system will be needed to resolve the chemical species present in the chemical signatures of humans to a degree where meaningful identifications of the individual components becomes possible. This work remains an objective for the future, and in many cases will require further enrichment of the sample to reach concentrations sufficient for identification.

The data were organized in a Kovats Index system and are tabulated in the Appendix.

The following conclusions were reached:

- (1) At the resolution available isothermally with 10-ft packed gas chromatographic columns and using collection and sample processing methods to ppb of organic species in air, the human chemical signatures exhibited from 32 to 48 features per signature.
- (2) Statistical pattern analysis conducted on the chemical signatures at the above resolution levels indicated that from 2 to 15 gas chromatographic variables are needed to reach meaningful classification of the signatures by their sources. The number of variables (features) that must be utilized increases as the similarity of sources (e.g. Caucasian males vs. females) or variability within classes of sources (e.g. males of several races vs. females of several races) increases.
- (3) Some classifications (e.g. humans vs. environments) based on classical stepwise discriminant analyses of patterns resulted in discriminant function which reasonably successfully classified additional test cases.
- (4) There were indications that the decision boundaries for the chemical signature classifications in statistical hyperspaces may be more complex than accessible to the classical stepwise discriminant procedures.
- (5) Detection and source discrimination devices based on the principle of chemical signatures must most probably be based on detection and correlation of 5 to 15 features at resolution levels outlined in the conclusion of paragraph #1.

- (6) Chromatographic analysis with resolution obtainable isothermally at several temperature levels from 200-ft support-coated Carbowax 20M column exhibited typically 100 to 130 peaks. Twenty-five of these, reanalyzed isothermally on 50-ft support-coated Apiezon L column separated further into 3 to 10 chemical components, thus producing over 150 observable components. The total number of components observable at this level of two-stage resolution in the chemical signature of a selected human, can be estimated to be in the range between 500 to 1000.
- (7) Classification of the observed components, by their gas chromatographic polarity, with attention to those components most frequently observed in humans, indicated the presence of considerable amounts of hydrocarbons, carbonyl compounds and a few alcohols. Compounds considerably more polar than alcohols were also observed and probably belong to species with more than two polar groups per molecule, or contain more than one double bond, in addition to a polar group.
- (8) Mass spectrographic identification succeeded only on two compounds which originate most probably from moth-proofing materials and therefore represent features characteristic of advanced material culture. The surprising frequency-of-occurrence of hydrocarbons may be related to a similar environment permeated by vehicular exhaust. Future mass spectrographic identification should be attempted only on components after their resolution in two-column system.

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Table I
EXAMPLES OF CALIBRATION FOR COLLECTION OF SELECTED COMPOUNDS
IN APIEZON L FLUIDIZED BED

Vapor	Detectability Limit With Present Technique, (molecules/cm ³)/cm ² ^a
n-Xylene	1.1 x 10 ¹⁰
n-Butanol	3.6 x 10 ¹⁰
n-Hexanol	5.2 x 10 ⁹
Octanone	7.2 x 10 ¹⁰
n-Heptaldehyde	6.1 x 10 ⁹
Acetic Acid	2.6 x 10 ¹¹
n-Propionic Acid	4.1 x 10 ¹⁰
n-Butyric Acid	5.7 x 10 ⁹
Ethyl Propionate	1.2 x 10 ¹¹
Ethyl Butyrate	6.1 x 10 ¹⁰
Dibutylamine	1.7 x 10 ¹¹

^aThe number of molecules per cubic centimeter of air that results in a 1-cm² peak at the highest sensitivity of the gas chromatograph

Table II
CALIBRATION OF TWO-COLUMN SYSTEM WITH n-ALKANES¹

n-Alkane ²	Elution Time, Min.		Corrected Retention Time, Min.	
	Carbowax 20M	Apiezon L	Carbowax 20M	Apiezon L
C ₈	4.74	5.83	0.71	0.73
C ₁₀	6.18	9.39	2.15	2.85
C ₁₁	7.77	13.68	3.74	5.55
C ₁₂	10.53	21.65	6.50	10.76
C ₁₃	15.15	36.42	11.12	20.91
C ₁₄	22.92	63.80	18.89	40.52
C ₁₅	37.24	118.36	33.21	80.76

¹The system at 125°C

²Elution data for methane: Carbowax 20M, 4.03 min; Apiezon L, 4.39 min

Table III
CALIBRATION OF TWO-COLUMN APPARATUS WITH POLAR COMPOUNDS¹

Compound ²	Corrected Retention Time, Min. ³		Kovats Retention Index	
	Carbowax 20M	Apiezon L	Carbowax 20M	Apiezon L
Ethanol	1.12	0.06	880	420
1-Propanol	2.18	0.12	1005	523
1-Butanol	4.15	0.30	1118	636
1-Pentanol	7.65	1.03	1235	726
Propionaldehyde	0.53	0.11	743	510
Butyraldehyde	1.00	0.20	859	600
Hexanal	3.53	0.67	1092	783
Heptanal	6.39	1.20	1198	870
Octanal	11.27	2.46	1304	976
2-Butanone	1.12	0.17	881	585
3-Pentanone	1.80	0.31	967	667
2-Heptanone	6.36	1.11	1197	859
2-Octanone	10.68	2.17	1290	958

¹The apparatus at 125°C

²Elution data for methane: Carbowax 20M, $M_C=4.03$ min; Apiezon L, $M_A=4.39$ min; $M_A-M_C=0.36$ min

³ $C_C=E_C-M_C$; $C_A=(E_A-E_C)-(M_A-M_C)$ where

C_C and C_A -Corrected retention times on Carbowax 20M and Apiezon L, in minutes, respectively

E_C and E_A -Elution times on Carbowax 20M and Apiezon L, in minutes, respectively

Table IV
CALIBRATION OF THREE-COLUMN APPARATUS WITH n-ALKANES¹

n-Alkane ²	Elution Time, Min.		Corrected Retention Time, Min.			Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L Short	Carbowax 20M	Apiezon L Short	Long	
C ₇	5.16	6.24	0.84	0.92	1.90	2.07
C ₉	8.13	12.56	4.02	4.27	8.83	2.07
C ₁₀	12.06	21.48	7.95	9.26	19.15	2.07
C ₁₁	19.20	39.33	15.09	19.99	39.98	2.00
C ₁₂	33.21	76.21 ⁴	29.10	43.00	88.00	2.05

¹The Apparatus at 90°C

²Methane elution data: Carbowax 20M, 4.11 min; Apiezon L-short, 4.27 min; Apiezon L-long, 4.44 min

³Average value, 2.05 ± 0.02

⁴From graphical extrapolation

Table V
CALIBRATION OF THREE-COLUMN APPARATUS WITH n-ALKANES¹

n-Alkane ²	Elution Time, Min.		Corrected Retention Time, Min.			Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L Short	Carbowax 20M	Apiezon L Short	Apiezon L Long	
C ₈	4.77	5.73	0.80	0.80	1.68	2.10
C ₉	5.40	7.10	1.43	1.54	3.19	2.07
C ₁₀	6.60	9.80	2.63	3.04	6.14	2.02
C ₁₁	8.61	14.54	4.64	5.77	11.75	2.02
C ₁₂	12.03	23.27	8.06	11.08	22.44	2.03
C ₁₃	17.76	39.93	13.79	22.00	42.95	1.95

¹The apparatus at 120°C

²Methane elution data: Carbowax 20M, 3.97 min; Apiezon L-short, 4.13 min;
Apiezon L-long, 4.27 min

³Average value, 2.03 ± 0.04

Table VI
CALIBRATION OF THREE-COLUMN APPARATUS WITH n-ALKANES¹

Compound ²	Elution Time, Min.			Corrected Retention Time, Min.			Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L		Carbowax 20M	Apiezon L		
		Short	Long		Short	Long	
C ₈	4.24	4.79	5.36	0.40	0.40	0.82	2.05
C ₉	4.50	5.35	6.23	0.66	0.70	1.43	2.04
C ₁₀	4.92	6.30	7.76	1.08	1.23	2.54	2.07
C ₁₁	5.61	8.03	10.48	1.77	2.27	4.57	2.01
C ₁₂	6.81	10.86	15.02	2.97	3.90	7.91	2.03
C ₁₃	8.64	15.63	22.94	4.80	6.84	14.00	2.05
C ₁₄	11.69	23.25	35.60	7.85	11.41	23.61	2.07

¹The apparatus at 150°C

²Methane elution data: Carbowax 20M, 3.84 min; Apiezon L-short, 3.99 min; Apiezon L-long, 4.14 min

³Average value, 2.04 ± 0.02

Table VII
CALIBRATION OF THREE-COLUMN APPARATUS WITH n-ALKANES¹

Compound ²	Elution Time, Min.			Corrected Retention Time, Min.			Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L		Carbowax 20M	Apiezon L		
		Short	Long		Short	Long	
C ₈	4.22	4.81	5.40	0.38	0.44	0.88	2.00
C ₉	4.48	5.38	6.28	0.64	0.75	1.50	2.00
C ₁₀	4.86	6.36	7.86	1.02	1.35	2.70	2.00
C ₁₁	5.64	8.09	10.59	1.80	2.30	4.65	2.02
C ₁₂	6.84	11.01	15.27	3.00	4.02	8.13	2.02
C ₁₃	8.67	15.83	23.12	4.83	7.01	14.15	2.02
C ₁₄	11.52	23.83	36.39	7.68	12.16	24.57	2.02

¹The apparatus at 150°C

²Methane elution data: Carbowax 20M, 3.84 min; Apiezon L-short, 3.99 min; Apiezon L-long, 4.14 min

³Average value, 2.01 ± 0.01

Table VIII
CALIBRATION OF THREE-COLUMN APPARATUS WITH ALDEHYDES¹

Compound ²	Elution Time, Min.		Corrected Retention Time, Min.		Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L Short Long	Carbowax 20M	Apiezon L Short Long	
Propionaldehyde	5.07	5.33 5.61	0.99	0.10 0.20	2.00
Butyraldehyde	5.67	6.01 6.39	1.59	0.18 0.38	2.11
Hexanal	8.94	9.79 10.74	4.86	0.69 1.46	2.11
Heptanal	12.36	13.88 15.55	8.28	1.36 2.85	2.09
Octanal	18.09	20.85 23.93	14.01	2.60 5.50	2.12

¹The apparatus at 120°C

²Elution data for methane: Carbowax 20M, 4.08 min; Apiezon L-short, 4.24 min; Apiezon L-long, 4.42 min

³Average value: 2.08 ± 0.04

Table IX
CALIBRATION OF THREE-COLUMN APPARATUS WITH n-ALCOHOLS¹

Compound ²	Elution Time, Min.		Corrected Retention Time, Min.				Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L Short	Apiezon L Long	Carbowax 20M	Apiezon L Short	Apiezon L Long	
Methanol	4.23	4.40	4.60	0.36	0.03	0.07	2.33
Ethanol	4.44	4.64	4.87	0.57	0.06	0.13	2.17
1-Propanol	4.89	5.15	5.44	1.02	0.12	0.25	2.08
1-Butanol	5.76	6.11	6.49	1.89	0.21	0.43	2.05
1-Pentanol	7.12	7.65	8.21	3.25	0.39	0.79	2.03
1-Hexanol	9.21	10.06	10.95	5.34	0.71	1.44	2.03

¹ System at 150°C

² Elution data for methane: Carbowax 20M, 3.87 min; Apiezon L-short, 4.01 min; Apiezon L-long, 4.17 min

³ Average value: 2.11 ± 0.09

Table X
CALIBRATION OF THREE-COLUMN APPARATUS WITH ALDEHYDES¹

Compound ²	Elution Time, Min.		Corrected Retention Time, Min.		Ratio ³ Apiezon L Long/Short
	Carbowax 20M	Apiezon L Short Long	Carbowax 20M	Apiezon L Short Long	
Propionaldehyde	4.26	4.46 4.68	0.40	0.06 0.12	2.18
Butyraldehyde	4.55	4.79 5.06	0.70	0.10 0.21	2.10
Hexanal	5.97	6.45 7.01	2.10	0.34 0.74	2.17
Heptanal	7.35	8.13 9.01	3.48	0.64 1.36	2.12
Octanal	9.48	10.86 12.28	5.61	1.24 2.50	2.02

¹System at 150 °C

²Elution data for methane: Carbowax 20M, 3.87 min; Apiezon L-short, 4.01 min; Apiezon L-long, 4.17 min

³Average value: 2.12 ± 0.06

Table XI
CONTRIBUTIONS TO EFFECTIVE CARBON NUMBER
IN HYDROGEN FLAME IONIZATION DETECTOR

Aliphatic C	1.0
Aromatic C	1.0
Olefinic C	0.95
Acetylenic C	1.30
Ketone, Aldehyde C	0
Nitrile C	0.3
Ether O	-1.0
Primary Alcohol O	-0.6
Secondary Alcohol O	-0.75
Tertiary Alcohol O	-0.25
Ester O	-0.25
Two or more Cl on Single	
Aliphatic C	-0.12 each Cl
Cl on Olefinic C	+0.05
Amine N	as O in corresponding alcohols

Table XII
n-BUTANOL EMISSION FROM VARIOUS HUMANS

Subject	Peak Area, cm ²	Rate Molecules/Sec
CAUCASIAN MALE		
A	2.00	0.62 x 10 ¹⁴
	1.60	0.49
	3.20	0.99
B ^a	7.78	2.42
	5.52	1.71
	3.28	1.02
	0.53	0.16
C ^b	1.76	0.55
	48.00	14.90
CAUCASIAN FEMALE		
D	11.64	3.61
E	6.08	1.89
F	46.30	14.40
INDIAN MALE		
G	3.60	1.12
H	4.13	1.28
I	3.84	1.19
J ^c	14.30	4.43
	4.00	1.24
	19.88	6.16
K	4.64	1.44
L	5.04	1.56
M	9.92	3.07
N	1.28	0.40
O	3.50	1.08
P	2.08	0.65
NEGRO MALE		
Q	5.28	1.64
NEGRO FEMALE		
R	60.48	18.75
S	56.40	

^aFirst 3 measurements taken when subject was on average mixed diet; 4th and 5th measurements taken 7 and 8 days later, respectively, after subject had been on special diet of fish, cheese, milk and tea.

^bSubject is a chemist working with various organic solvents.

^cSubject is a strict vegetarian; other Indian subjects mostly vegetarians.

Table XIII
DISCRIMINANT ANALYSIS OF CHEMICAL SIGNATURES
OF MALES, FEMALES, AND AIR FROM VARIOUS ENVIRONMENTS

Consecutive Number*	Variables Considered	Number of Cases Considered		
	Corresponding Kovats Index Range	Males, 39	Females, 26	Air, 10
Cases Correctly Classified, %				
1	1250-1300	77	34	70
2	1350-1400	77	38	60
3	1450-1500	82	51	90
4	1050-1100	84	48	90
5	650- 700	84	62	90
6	> 1600	84	62	90
.				
.				
.				
.				
15	850- 900	87	79	90

* In order selected by the computer

At 15 variables considered, the following Kovats Index ranges have not been selected by the computer as useful discriminants: 700-750; 800-850; 950-1000; 1000-1050; 1150-1200; 1400-1450; 1550-1600.

Table XIV
DISCRIMINANT ANALYSIS OF CHEMICAL SIGNATURES
OF CAUCASIAN MALES, INDIAN MALES, AND CAUCASIAN FEMALES

Consecutive Number*	Variables Considered Corresponding Kovats Index Range	Number of Cases Considered		
		Caucasian Males, 25	Indian Males, 12	Caucasian Females, 23
		Cases Correctly Classified, %		
1	1250-1300	60	41	61
2	700- 750	56	75	73
3	1350-1400	64	75	69
4	650- 700	64	75	69
5	1550-1600	72	75	69
.				
.				
.				
13	> 1600	83	83	80

* In order selected by the computer

At 13 variables considered, the following Kovats Index ranges have not been selected by the computer as useful discriminants:
< 600; 600-650; 850-900; 900-950; 1150-1200; 1200-1250;
1300-1350; 1400-1450; 1500-1550.

Table XV
LIST OF VARIABLES (K.I. RANGES)
(Those ranges in which peaks most frequently
occurred in vapor from humans are marked P)

Consecutive Number of Range, (Variable)	K.I. Range From-To	Range Width, K.I. Units	Variables Selected for Latest Class Separations
1	0- 465	465	
2	465- 485	20 P	
3	485- 516	31	
4	516- 536	20 P	x P
5	536- 590	54	x
6	590- 610	20 P	x P
7	610- 645	35	
8	645- 665	20 P	x P
9	665- 666	1	
10	666- 686	20 P	x P
11	686- 706	20 P	x P
12	706- 722	16	
13	722- 740	18 P	
14	740- 758	18 P	
15	758- 799	41	x
16	799- 819	20 P	
17	819- 824	5	x
18	824- 842	18 P	x P
19	842- 860	18 P	
20	860- 882	22	
21	882- 902	20 P	
22	902- 904	2	x
23	904- 924	20 P	x P
24	924- 968	44	
25	968- 987	19 P	
26	987-1005	18 P	x P
27	1005-1025	20	x
28	1025-1045	20 P	
29	1045-1061	16	
30	1061-1081	20 P	x P
31	1081-1084	3	
32	1084-1104	20 P	
33	1104-1112	8	x
34	1112-1132	20 P	
35	1132-1140	8	x
36	1140-1160	20 P	
37	1160-1171	11	x
38	1171-1191	20 P	
39	1191-1195	4	
40	1195-1215	20 P	
41	1215-1232	17 P	
42	1232-1250	18 P	
43	1250-1265	15	x
44	1265-1285	20 P	
45	1285-1297	12	
46	1297-1315	18 P	x P
47	1315-1334	19 P	x P
48	1334-1340	6	
49	1340-1360	20 P	x P
50	1360-1372	12	x
51	1372-1392	20 P	x P
52	1392-1401	9	x
53	1401-1419	18 P	
54	1419-1437	17 P	x P
55	1437-1454	17	x
56	1454-1472	18 P	x P
57	1472-1490	18 P	x P
58	1490-1500	10	x
59	1500-1517	17 P	x
60	1517-1535	18 P	x P
61	1535-1590	55	x
62	1590-1610	20 P	
63	1610-1615	5	x
64	1615-1635	20 P	
65	1635-1800	165	x

Table XVI
 SUCCESS IN STATISTICAL PATTERN CLASSIFICATION
 AS A FUNCTION OF THE NUMBER OF VARIABLES
 (Variables Correspond to Range Numbers in Table XV)

Consecutive Numbering	<u>All 37 P-Ranges (Run A)</u>		<u>10 P-Ranges Only (Run B)</u>	
	<u>Identification Number of Variable of Table XV</u>	<u>% of Human Signatures Classified in "Human Class"</u>	<u>Identification Number of Variable of Table XV</u>	<u>% of Human Signatures Classified in "Human Class"</u>
1	64	71	64	71
2	56	78	56	78
3	57	83	57	83
4	47	83	26	87
5	46	83	44	91
6	26	86		
7	18	86		
8	10	no readout		
9	30	88		
10	8	no readout		
11	16	94		
Additional		92 to 94		

Table XVII

SEVERAL IDENTIFICATIONS OF COMPONENTS IN CHEMICAL SIGNATURE
FROM HUMAN SUBJECT BY THREE-POINT MATCH
IN THREE-COLUMN APPARATUS¹

Compound	E l u t i o n T i m e , M i n .					
	Carbowax 20M		Apiezon L-Short		Apiezon L-Long	
	Calibration	Signature ²	Calibration	Signature ²	Calibration	Signature ²
Methanol	4.23	4.29	4.40	4.48	4.60	4.68
Ethanol	4.44	4.38	4.64	4.63	4.87	4.86
Butyraldehyde	4.55	4.53	4.79	4.77	5.06	5.05
Heptanal	7.35	7.53	8.13	8.27	9.01	9.03
Octanal	9.48	9.48	10.86	10.68	12.28	12.45

¹The apparatus at 150°C

²Human subject: Caucasian female. L.B.

Table XVIII
CHARACTERIZATION OF HUMAN CHEMICAL SIGNATURE COMPONENTS ON TWO-COLUMN GAS CHROMATOGRAPHIC APPARATUS¹

Peak Examined Consecutive Number	Highest Occurrence Frequency Peaks in Human Chemical Signatures Kovats Index Range Carbowax 20M, 125°C	Component Characterization										Approximate Boiling Point, °C	Closest Tentative Identification
		C - I, 20M - 1 Apl											
		Kovats Index Carbowax 20M	Apiezon L2	0 - 100	100 - 250	250 - 380	380 - 520	520 - 563					
1	722- 740, 29P	715	463* 567 615		142 100	252*						50 70 80	(methylal)
2	739- 819, 45P	810	545* 440 656 688 795 822		154 122	370 265*						60 50 100 90 110 120	(acetone) (octane)
3	882- 902, 45P	879	545* 902* 316 657 718 865	-23*	222 161	134*		563				75 140 70 90 105 130	(butyral) (nonane) (methanol)
4	882- 902, 45P	877	545* 899* 393 640 656	-22*	237 221	332*		484				80 140 80 90 100	(butyral) (nonane) (ethanol)
5	968- 987, 48P	985	633* 990* 645 775 795 953 965 985	-5*	210 200	352*						90 160 110 120 120 150 150 160	(pentanal)
6	987-1005, 67P	1000	1002* 525* 697 850 930 945 965 975	-2*	150	475*						160 100 120 140 145 150 155 160	(propanol)
7	1025-1045, 34P	1030	545 697 1005	25		333		486				110 115 165	

Table XVIII (continued)
CHARACTERIZATION OF HUMAN CHEMICAL SIGNATURE COMPONENTS ON TWO-COLUMN GAS CHROMATOGRAPHIC APPARATUS¹

Peak Examined Consecutive Number	Highest Occurrence Frequency Peaks in Human Chemical Signature Kovats Index Range Carbowax 20M/125°C	Component Characterization										Approximate Boiling Point, °C	Closest Tentative Identification
		L = IC20M-1Apl											
		Kovats Index											
		Carbowax 20M	Ablezon 12	0 - 100	100 - 250	250 - 380	380 - 520	> 520					
8	1025-1045, 34P	1050	620* 715* 1060* 775 822 850 859 966 988	-10*		335*	430*					120 125 180 130 140 145 150 155 160	
						275							
					228 200 192								
				84 62									
9	1061-1081, 48P	1078	783* 1100* 828 1054 1065 1084 1093	-22*		295*						130 185 140 170 175 175 180	(hexanal) (undecane)
						250							
10	1084-1104, 45P	1095	747* 1100* 577 718 813 1050 1062 1083 1090	-5*		348*						125 180 110 120 130 130 175 180 180 180	
11	1084-1104, 45P	1103	630* 1103* 936 865 1050 1062 1085	0*		267	473*					120 180 145 145 175 180 180	(undecane)
					238								
12	1215-1232, 47P	1220	987* 865 903 947 1055			355 317 273	233*					175 150 160 170 185	(heptanal)

Table XVII (continued)
CHARACTERIZATION OF HUMAN CHEMICAL SIGNATURE COMPONENTS ON TWO-COLUMN GAS CHROMATOGRAPHIC APPARATUS¹

Peak Examined Consecutive Number	Highest Occurrence Frequency Peak in Human Chemical Signature Kovats Index Range Carbowax 20M, 125°C	C o m p o n e n t C h a r a c t e r i z a t i o n										Approximate Boiling Point, °C	Closest Tentative Identification
		I C 20M-1 Ap1											
		Kovats Index Carbowax 20M	Apiezon L4	0 - 100	100 - 250	250 - 380	380 - 520	520 - 520					
13	1265-1285, 60P	1275	1014* 883 914 977 1037 1078				261*	392				180 160 170 175 180 190	
14	1297-1315, 54P	1292	977* 1018* 907 1010		238 217		315* 254*	385				170 180 175 180	(octanal)
15	1297-1315, 54P	1292	974* 760 980 992 1044 1140 1180				318*		532			170 > 140 180 180 180 205 210	(octanal)
16	1297-1315, 54P	1295	972* 803 970 950 1003 1045 1070 1140		248 152 112		323*	402				175 170 170 170 175 180 190 205	
17	1340-1360, 64P	1345	950* 975 980 1032 1078 1115		225 155			395* 420				170 175 180 185 190 195	
18	1372-1392, 65P	1383	965* 1390* 995 1035 1052 1114	-7*				418*	388	348 331 269		180 240 180 180 190 210	

Table XVIII (continued)
CHARACTERIZATION OF HUMAN CHEMICAL SIGNATURE COMPONENTS ON TWO-COLUMN GAS CHROMATOGRAPHIC APPARATUS¹

Peak Examined Consecutive Number	Highest Occurrence Frequency Peaks in Human Chemical Signatures Kovats Index Range Carbowax 20M, 125°C	Component Characterization .. = 1 C20M ⁻¹ ApI										Approximate Boiling Point, °C	Closest Tentative Identification
		Kovats Index											
		Carbowax 20M	Apiezon L2	0 - 100	100 - 250	250 - 380	380 - 520	520 - 720	720 - 920	920 - 1120	1120 - 1320		
		Apiezon L2	0 - 100	100 - 250	250 - 380	380 - 520	520 - 720	720 - 920	920 - 1120	1120 - 1320			
19	1372-1392, 65P	1390	1075* 1235* 753 1058 1093		155*			637			190 225 > 150 190 200		
20	1372-1392, 65P	1392	1077* 635 1054 1120 1148 1200 1265 1390 1398			315* 338 272 244 192 127		757			195 > 130 185 210 210 225		
21	1419-1437, 50P	1443	1162* 946 1068 1124 1185	2 -6		281* 375 319 258	497				210 180 195 210 220	(tetradecane) (heptanol)	
22	1454-172, 69P	1468	1153* 734 923 1015 1080 1123			315* 346		734 545	453 388		220 > 150 > 180 195 210 190		
23	1472-1490, 66P	1490	1192* 735 1003 1077 1162			298* 328		755			220 > 150 190 205 215		
24	1472-1490, 66P	1493	702* 1177* 897 980 1012 1075 1150 1187 1195 1213			316* 343 306 298 280		791* 606			> 150 220 > 170 185 195 205 220 225 225 230		

Table XVIII (continued)
CHARACTERIZATION OF HUMAN CHEMICAL SIGNATURE COMPONENTS ON TWO-COLUMN GAS CHROMATOGRAPHIC APPARATUS¹

Peak Examined Consecutive Number	Highest Occurrence Frequency Peaks in Human Chemical Signature Kovats Index Range Carbowax 20M, 125°C	Component Characterization					Approximate Boiling Point, °C	Closest Tentative Identification
		Kovats Index Carbowax 20M	Apiezon L ²	0 - 100	100 - 250	250 - 360	360 - 520	520 - 785
25	1517-1515, 63P	1540	765					150
			1150					220
			1170				390	225
			1187			370		225
			1273			353		240
			1305		190	267		240

¹Four signature collections from the same Caucasian female, L.B.

²The asterisk designates major signature components.

³The letter "p" following the number designates "peak".

⁴Boiling points estimated from Apiezon L Kovats Index using plot in Advances in Gas Chromatography 1, 218 (1965).

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20	Comparison in the Improvement of Two-Way Classification of Chemical Signatures: Using Variables that Frequently Contain Peaks in Human Signatures or Variables that Correspond to Ranges Inbetween

LIST OF FIGURES (continued)

<u>Figure Number</u>	<u>Title</u>
21	Derivation of Discriminant Function for Classification of Human and Environment Chemical Signatures and Test of the Function on 29 Additional Human Chemical Signatures
22	Improvement in Correct Pattern Assignments with Increase in the Number of Variables Utilized by the Stepwise Discriminant Analysis Program
23	Derivation of Three-Class Discriminant Functions Field and Test of the Derived Functions with Additional Male Chemical Signature Patterns
24	Three-Column Data Interpretation Assisted by GA3COL Computer Program
25	Display of Two-Column Data on Human Chemical Signatures

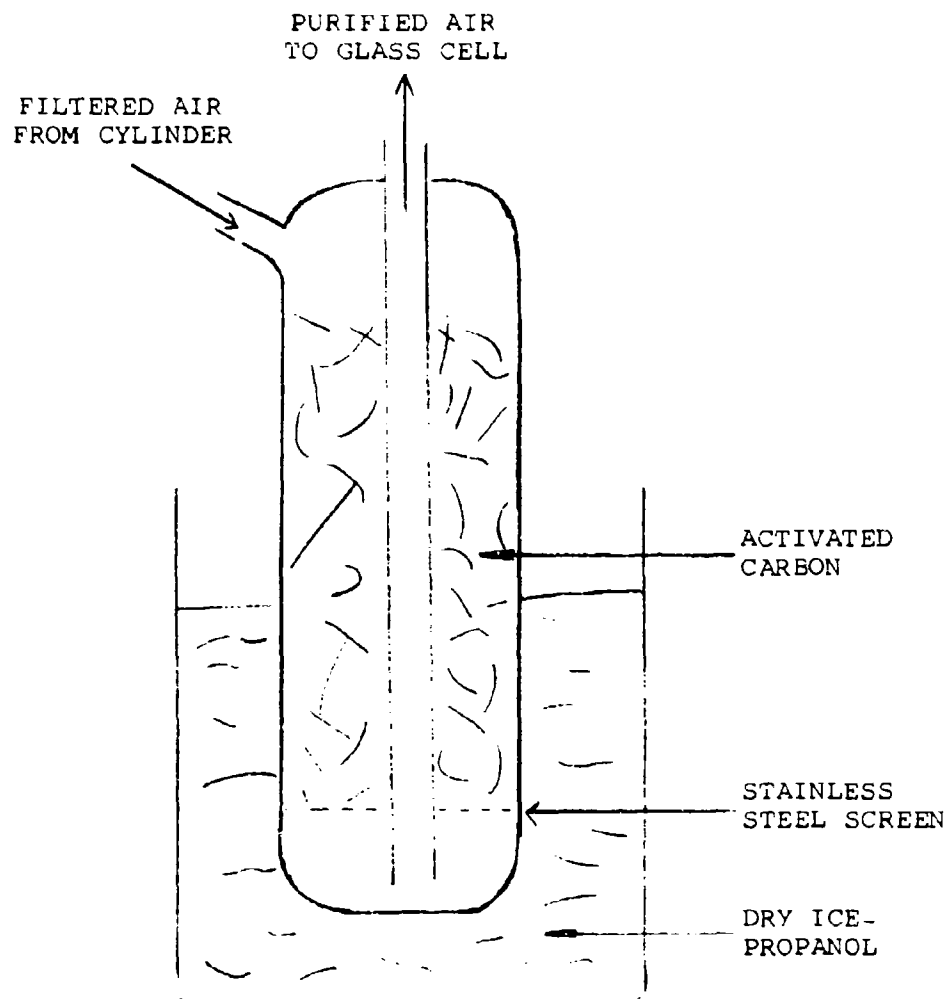


Figure I
CARBON BED FOR REMOVAL OF RESIDUAL ORGANIC VAPOR
FROM HOSPITAL GRADE CYLINDER AIR

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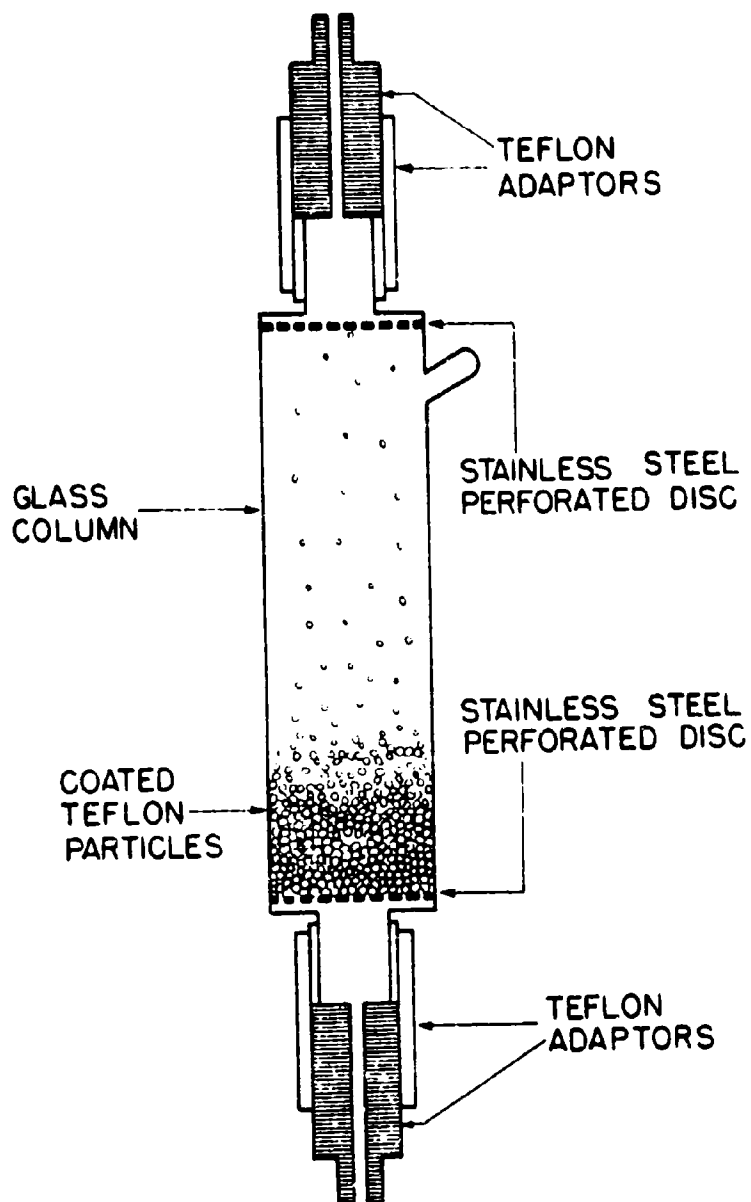


Figure 2
FLUIDIZED BED SAMPLER

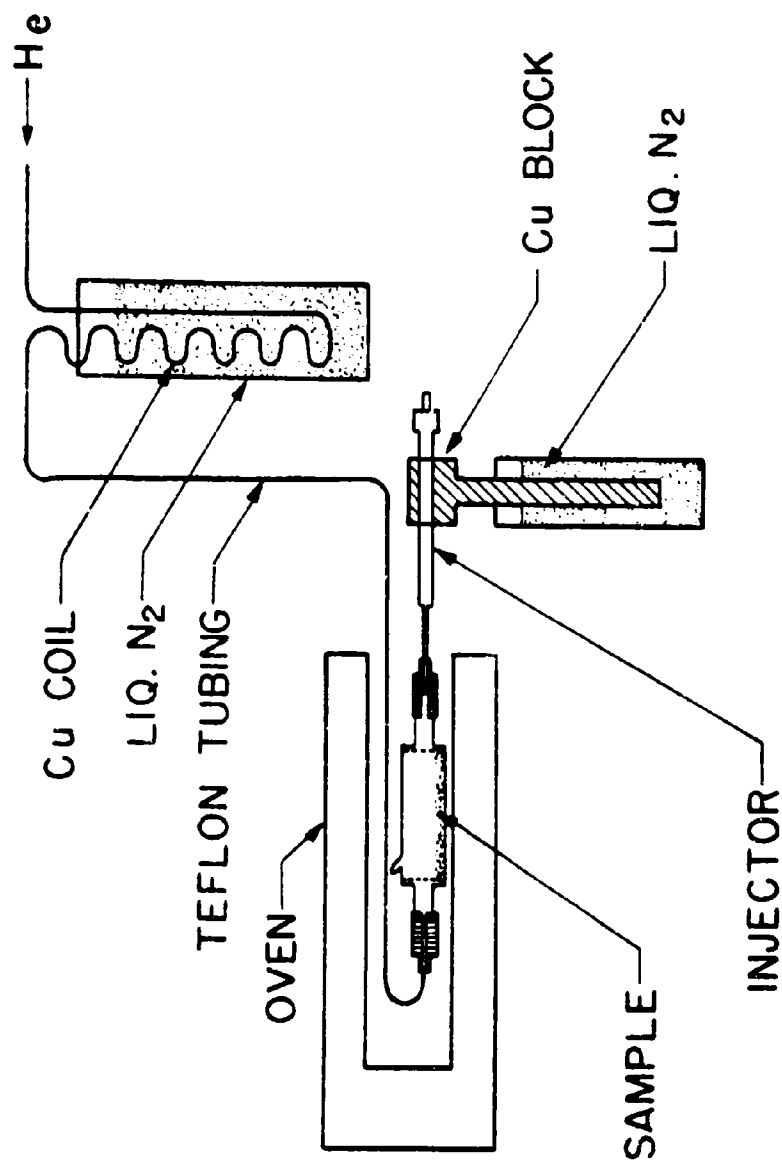
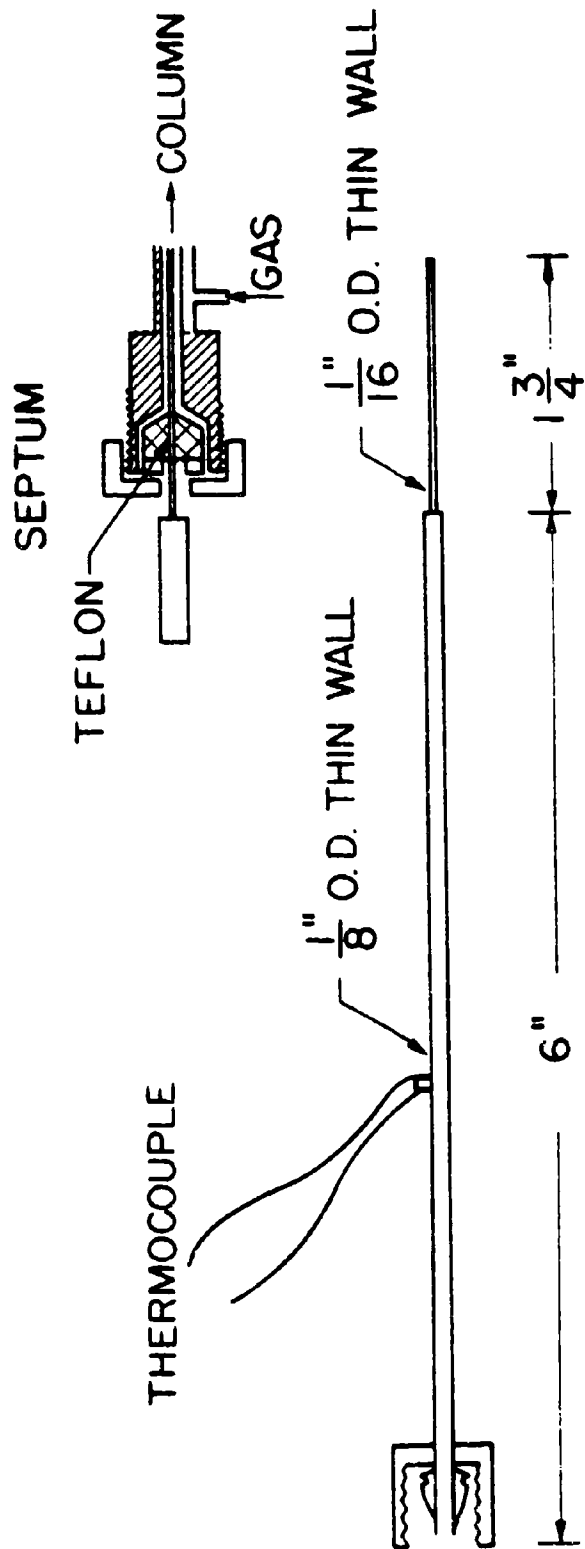


Figure 3
TRANSFER OF SAMPLE FROM BED SAMPLER TO INJECTOR



(Material : Stainless Steel)

Figure 4
SAMPLE INJECTOR

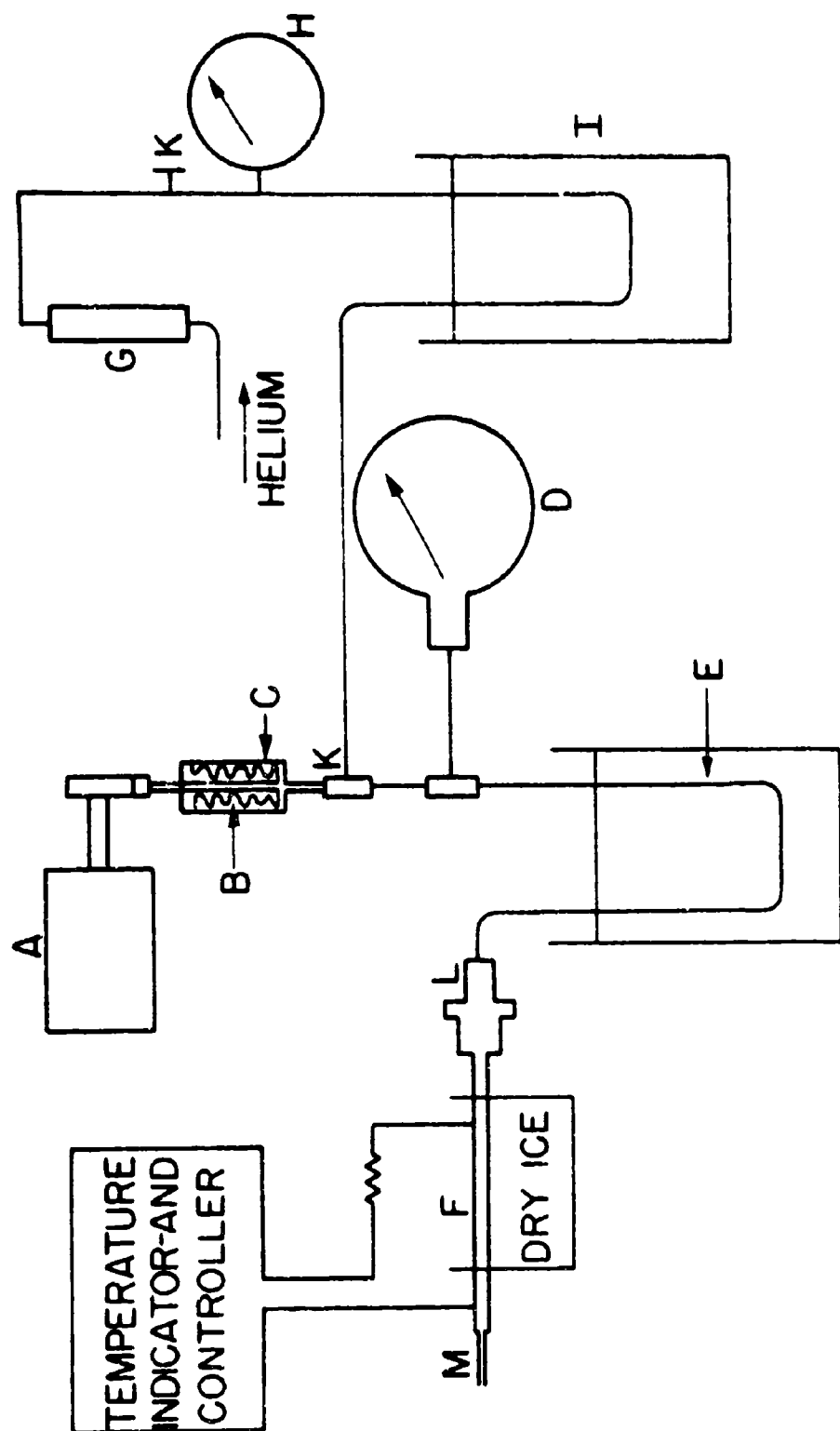


Figure 5
SAMPLE INJECTION SYSTEM

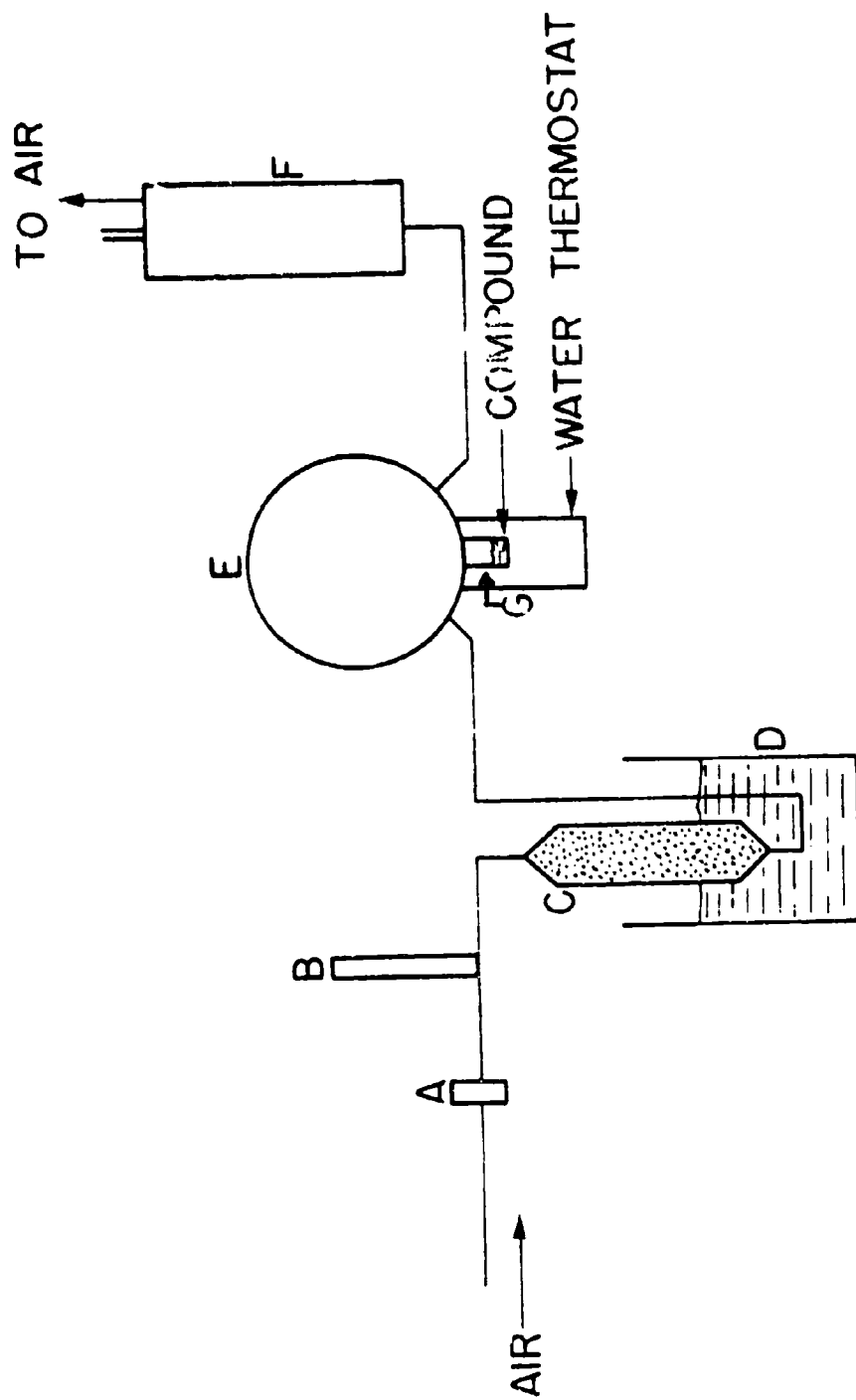


Figure 6
CALIBRATION SYSTEM

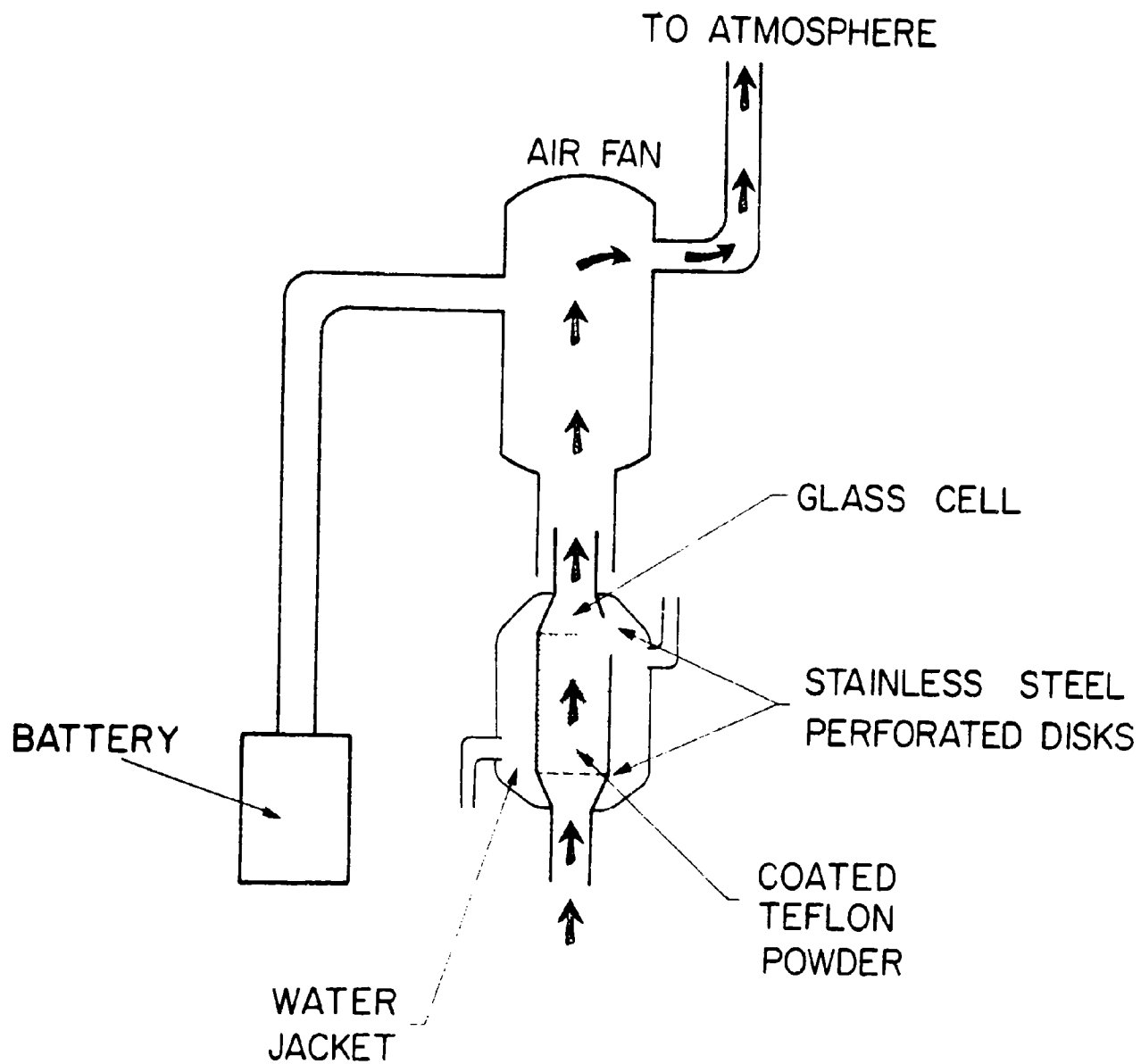


Figure 7
PORTABLE AIR SAMPLING APPARATUS

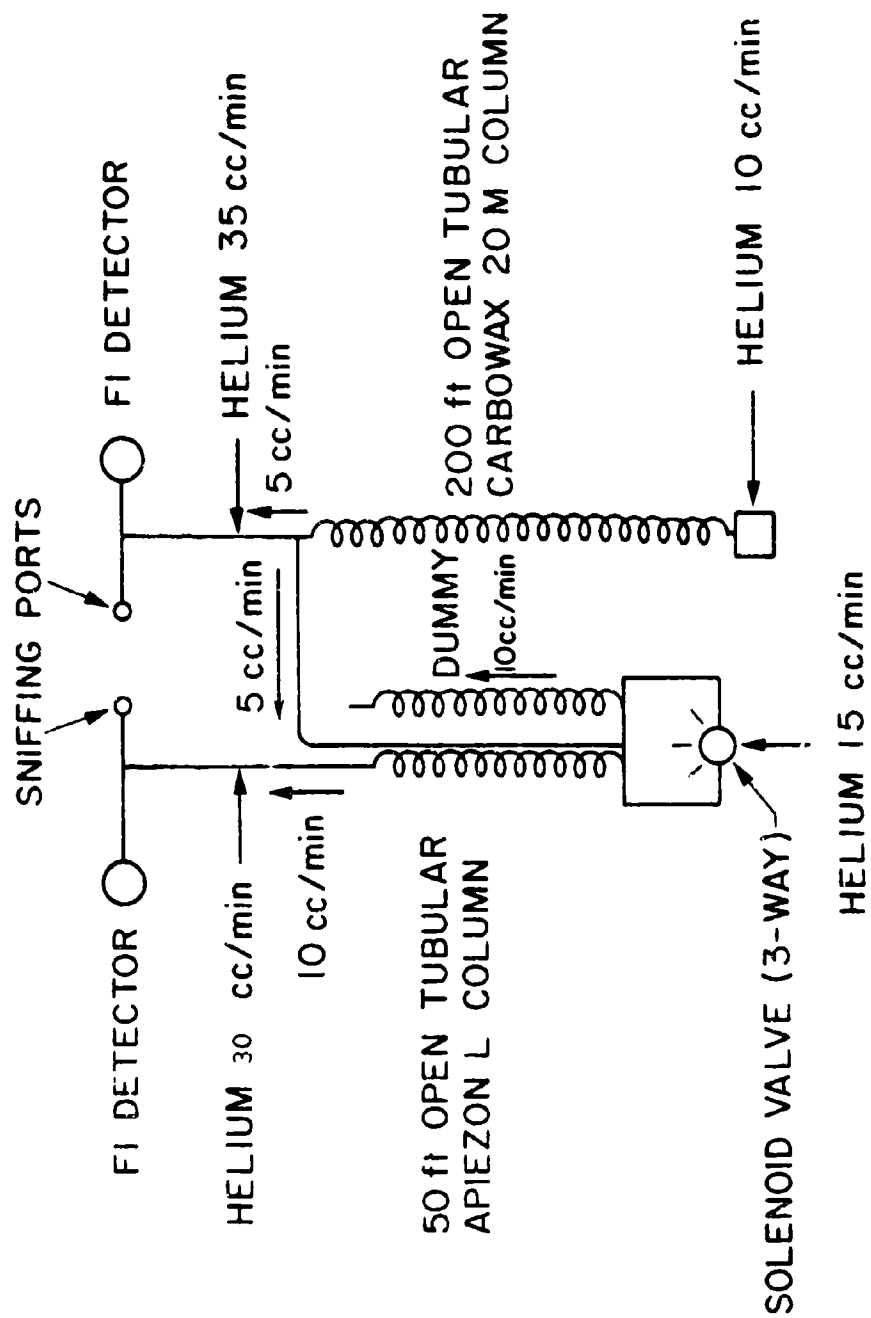


Figure 8
TWO-COLUMN APPARATUS

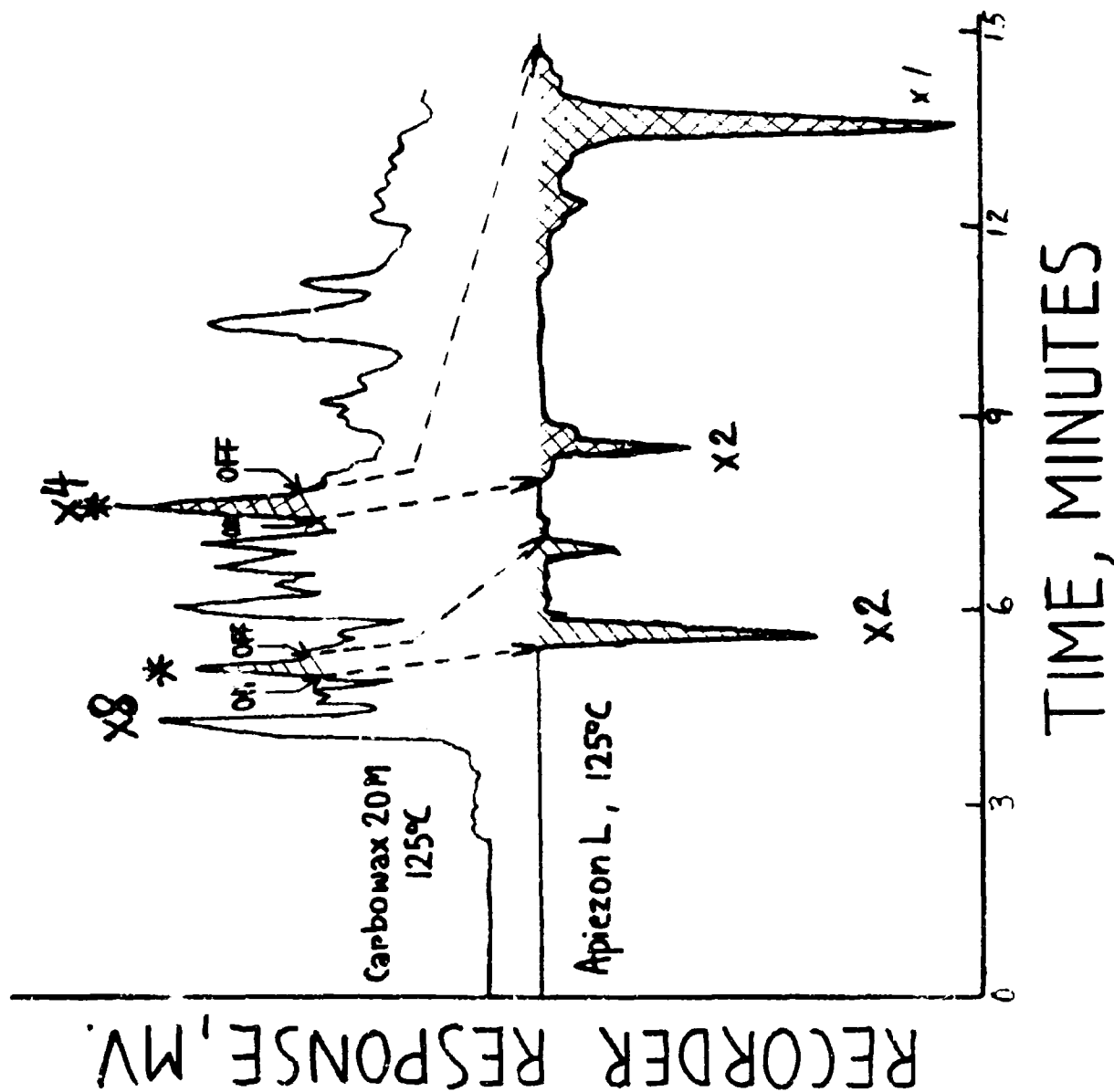


Figure 9
SECONDARY RESOLUTION OF TWO HUMAN SIGNATURE COMPONENTS
ON APIEZON L COLUMN OF TWO-COLUMN APPARATUS

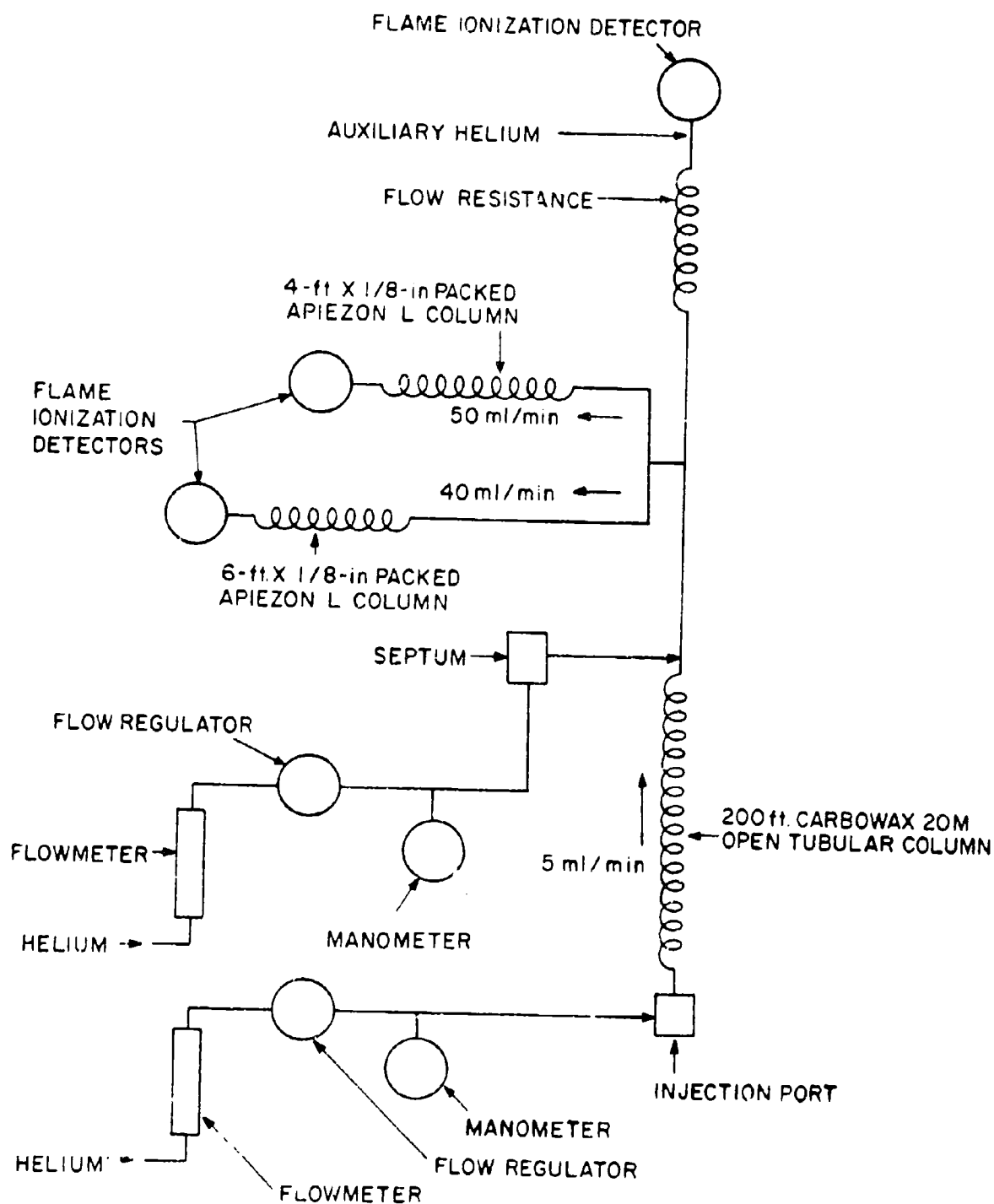


Figure 10
THREE COLUMN APPARATUS

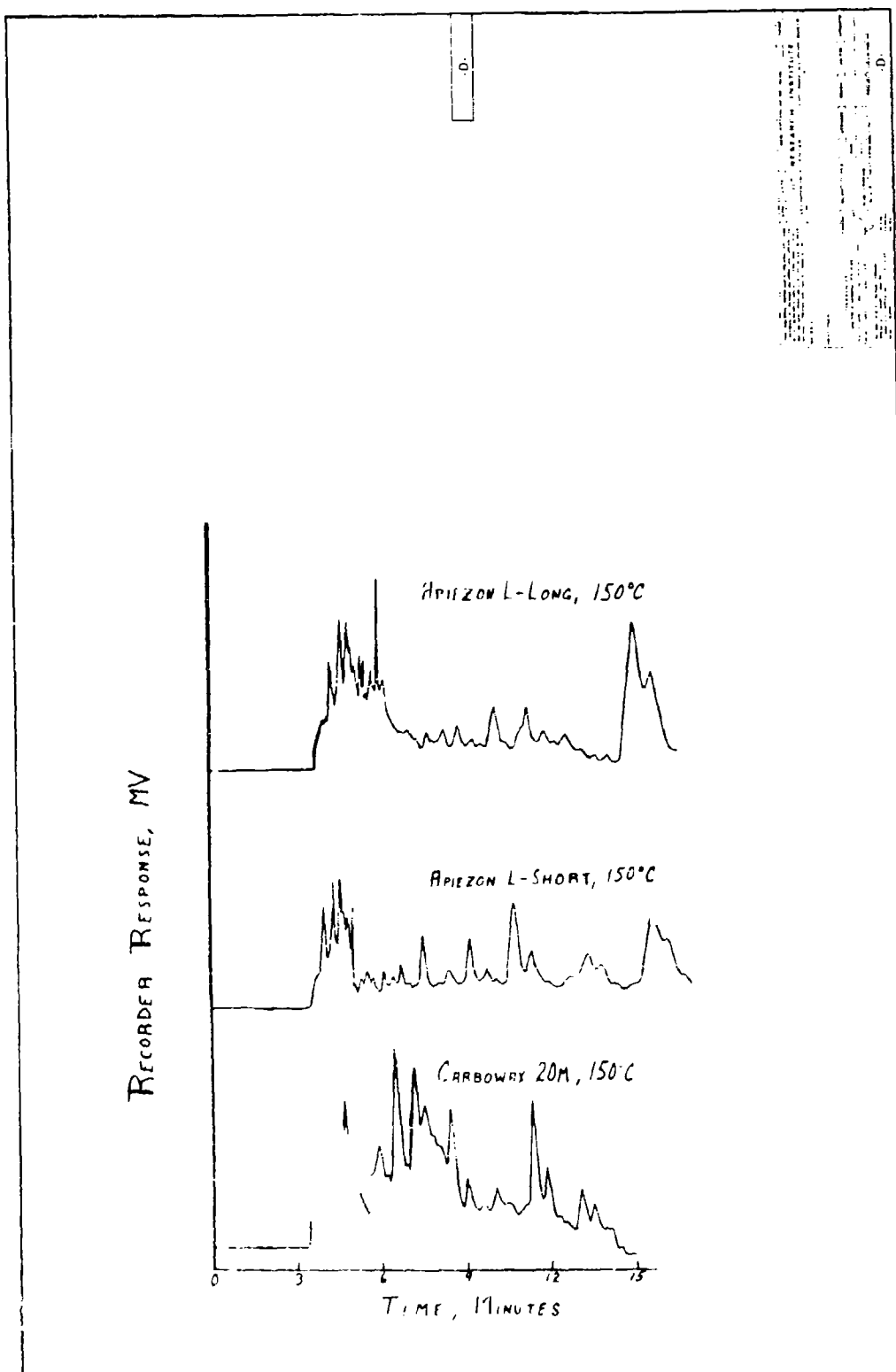


Figure 11
A SEGMENT OF A TYPICAL GAS CHROMATOGRAM OF
HUMAN CHEMICAL SIGNATURE OBTAINED ON THREE-COLUMN APPARATUS

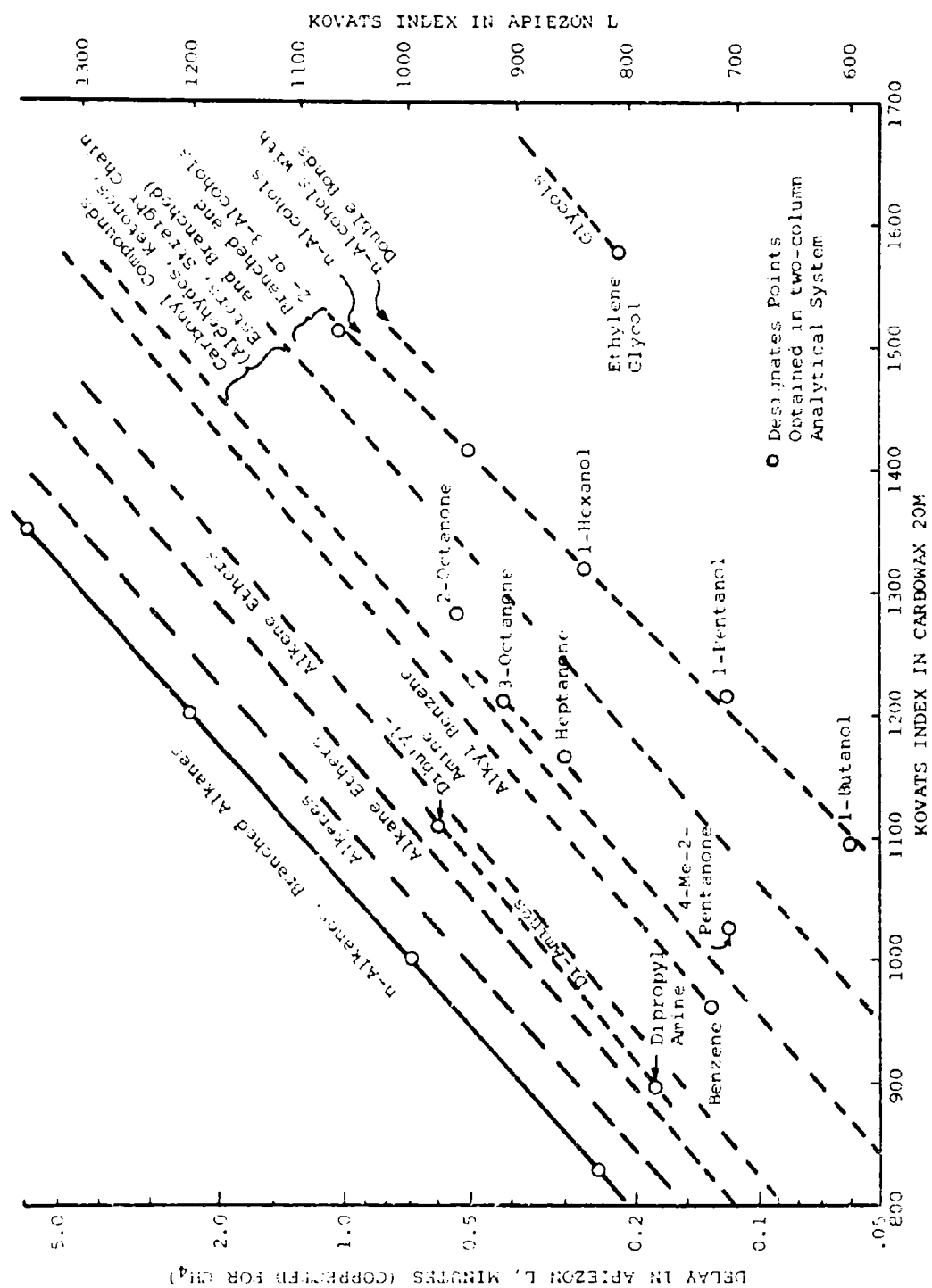


Figure 12
CHEMICAL LOGIC FIELD FOR CLASSIFICATION OF DATA OBTAINED
FROM THE TWO-COLUMN SYSTEM

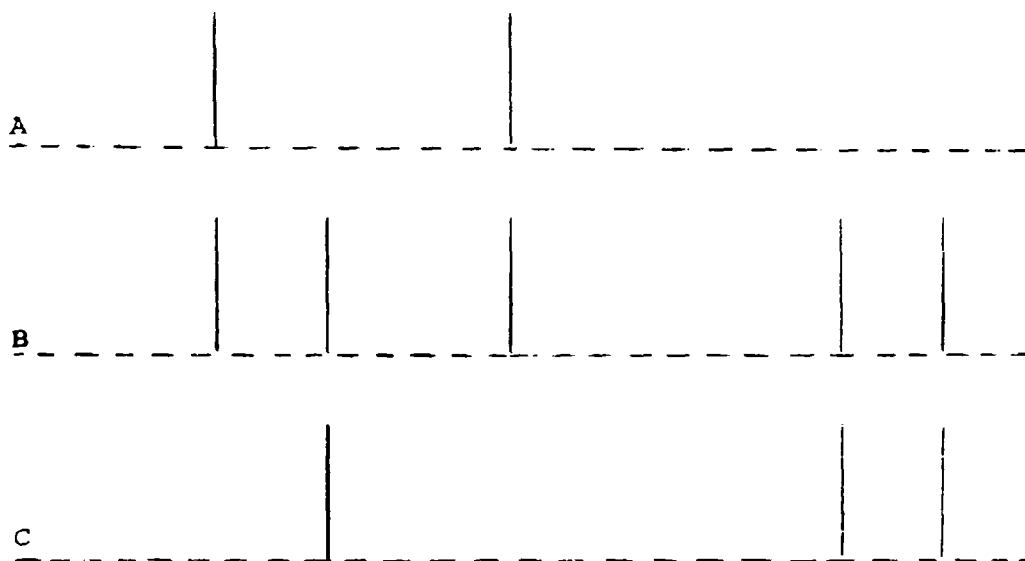


Figure 13
PROBLEMS IN PATTERN SIMILARITY COMPARISONS

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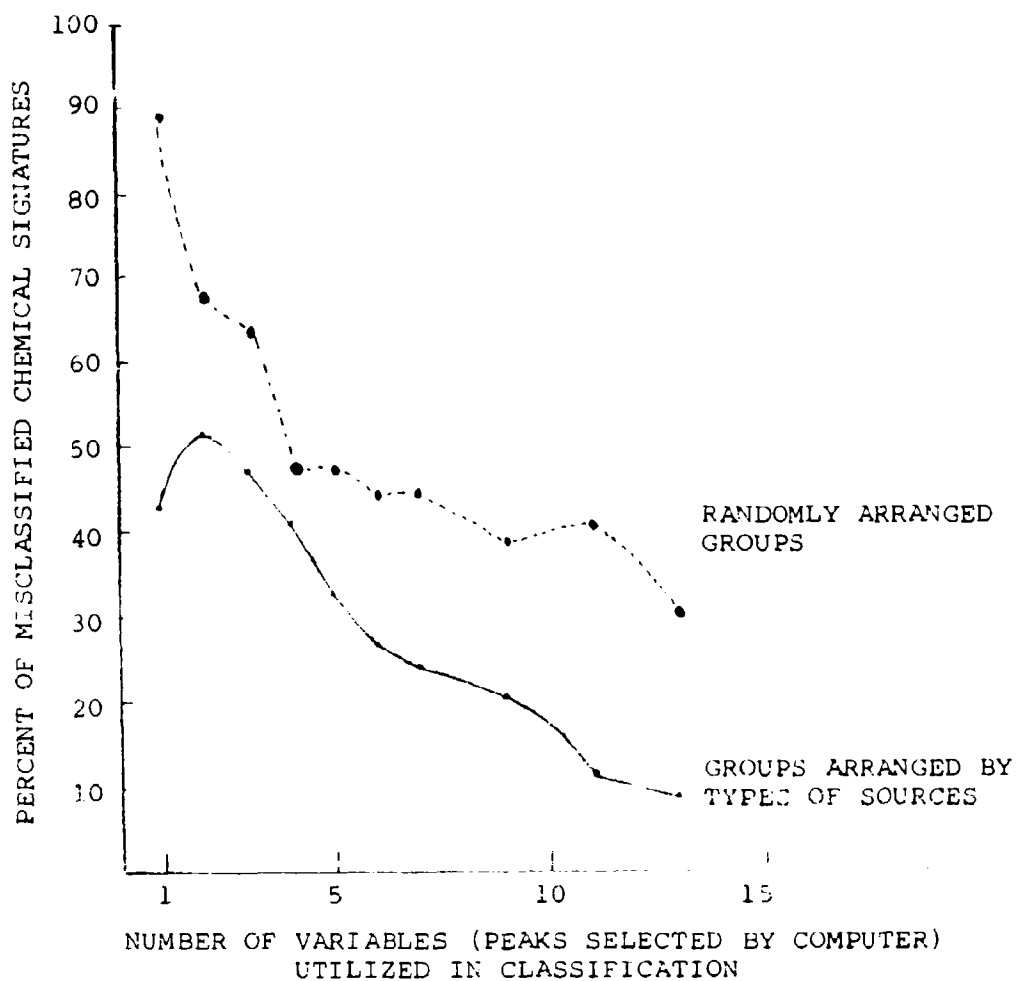


Figure 14

IMPROVEMENT IN CORRECT CLASSIFICATION OF INDIVIDUAL
CHEMICAL SIGNATURES WITH INCREASE IN THE NUMBER OF COMPUTER-
SELECTED PEAKS EMPLOYED IN THE CLASSIFICATION

Classification based on 49 signatures arranged in 5 groups.

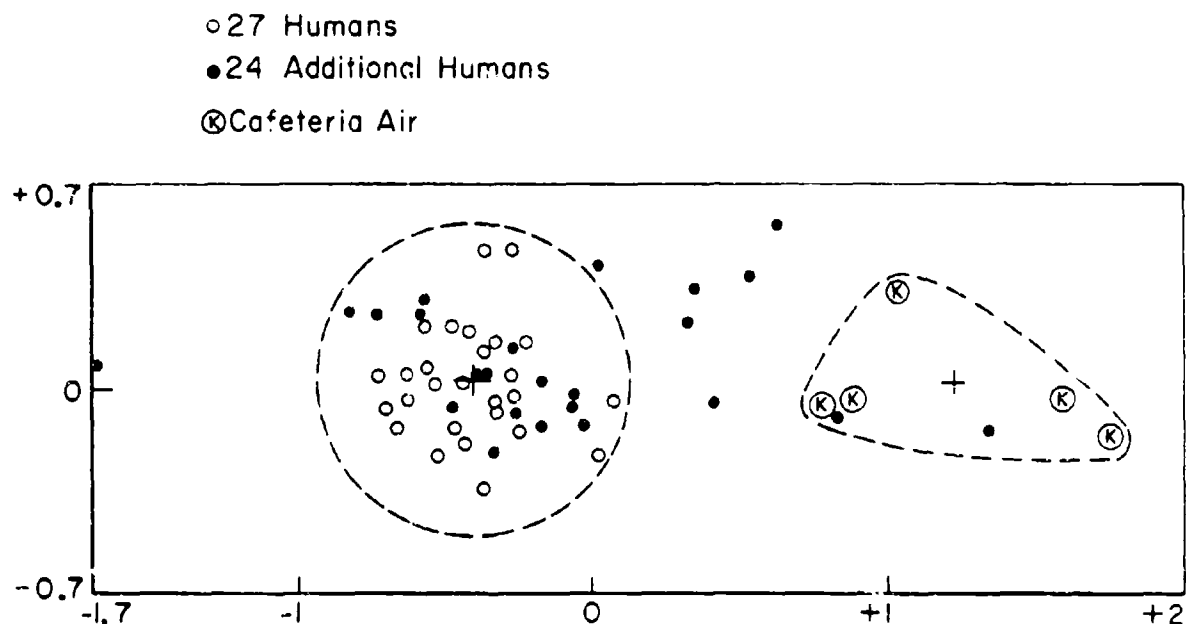
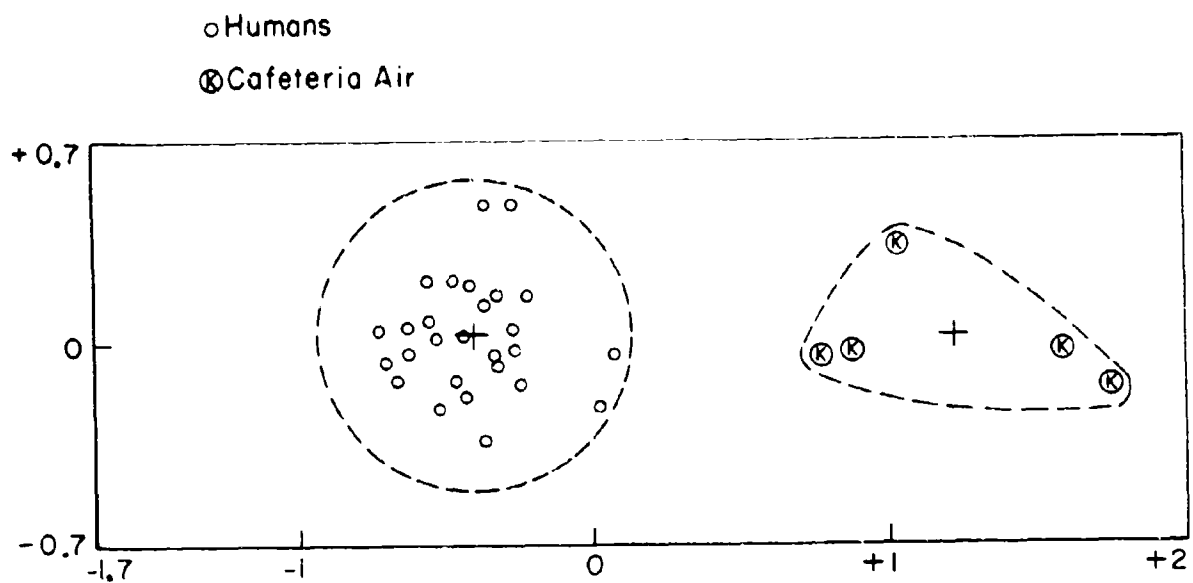


Figure 15
DISCRIMINANT FUNCTIONS FIELD FOR HUMANS VS. COOKING AREA VAPORS
AND CLASSIFICATION OF ADDITIONAL HUMANS USING DERIVED FUNCTIONS

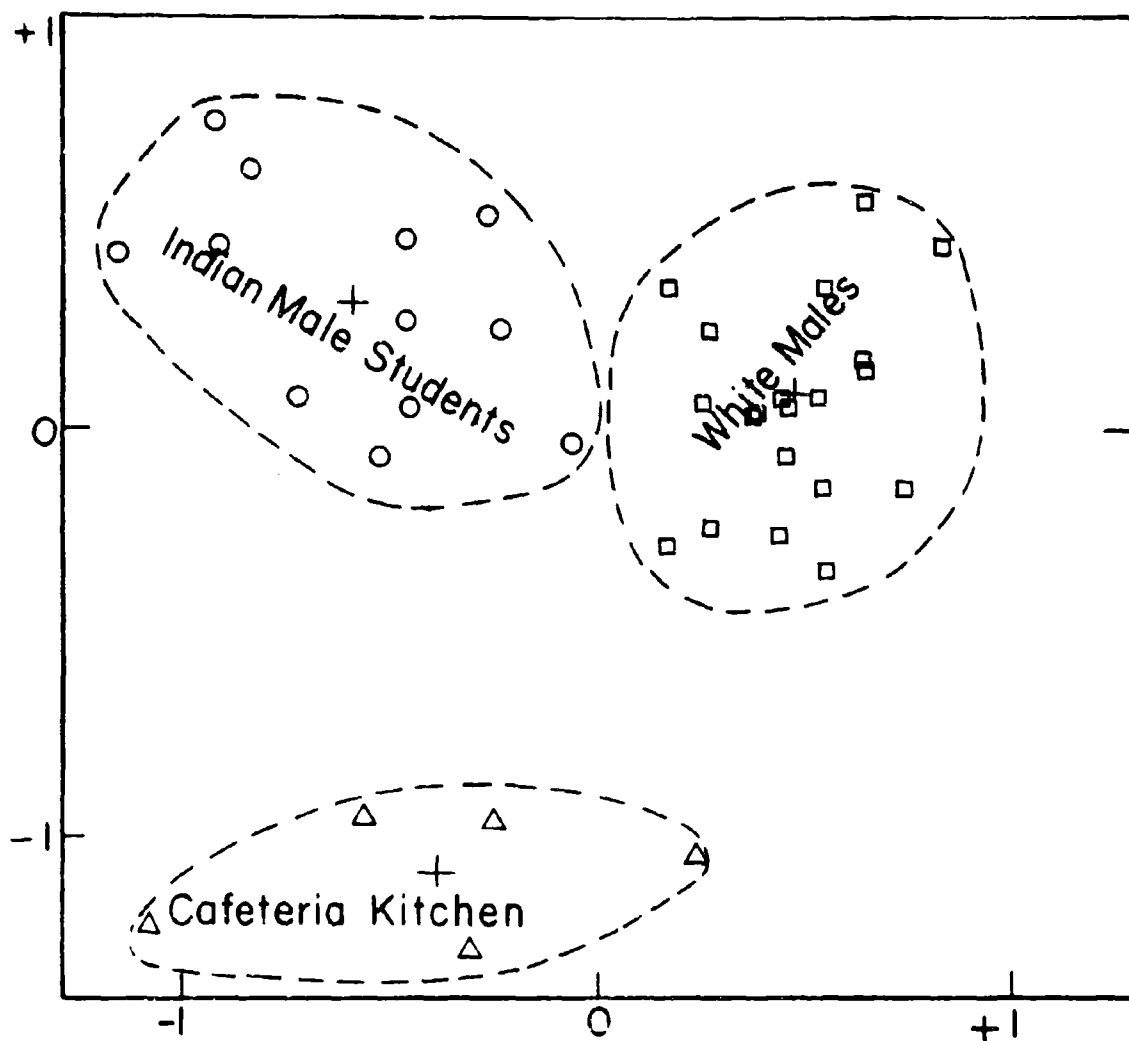


Figure 16
 STATISTICAL CLASSIFICATION PLANE FOR VAPOR
 CHROMATOGRAMS OF TWO TYPES OF MALES AND FOOD PREPARATION AREA

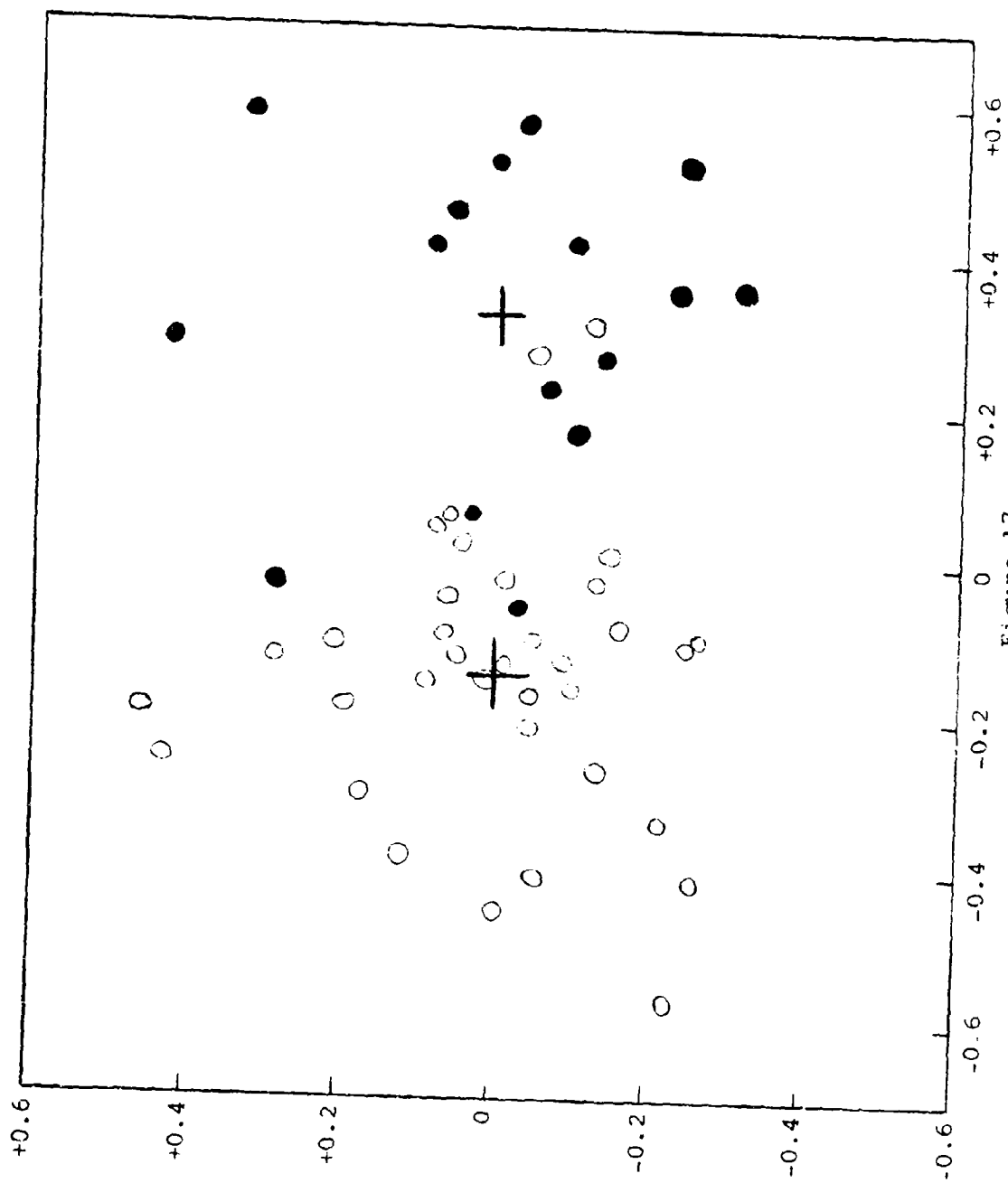


Figure 17

CLASSIFICATION FIELD FOR GLASS CELL EXPERIMENTS ONLY

Subjects: 35 males, 16 females

Variables: Input, 21. Utilized: 7. Crosses indicate the centers for groups

NOTES TO FIGURE 17

Wilcoxon-White ranking test, applied to the positions of points in the negative direction of X discriminant function only yielded 162 as the rank sum for females (rank sum for the smallest group). The critical point for error probability of 0.1 percent is 252. Thus, the validity of the X function in classification of males versus females has considerably less than 1:1000 chance of being in error.

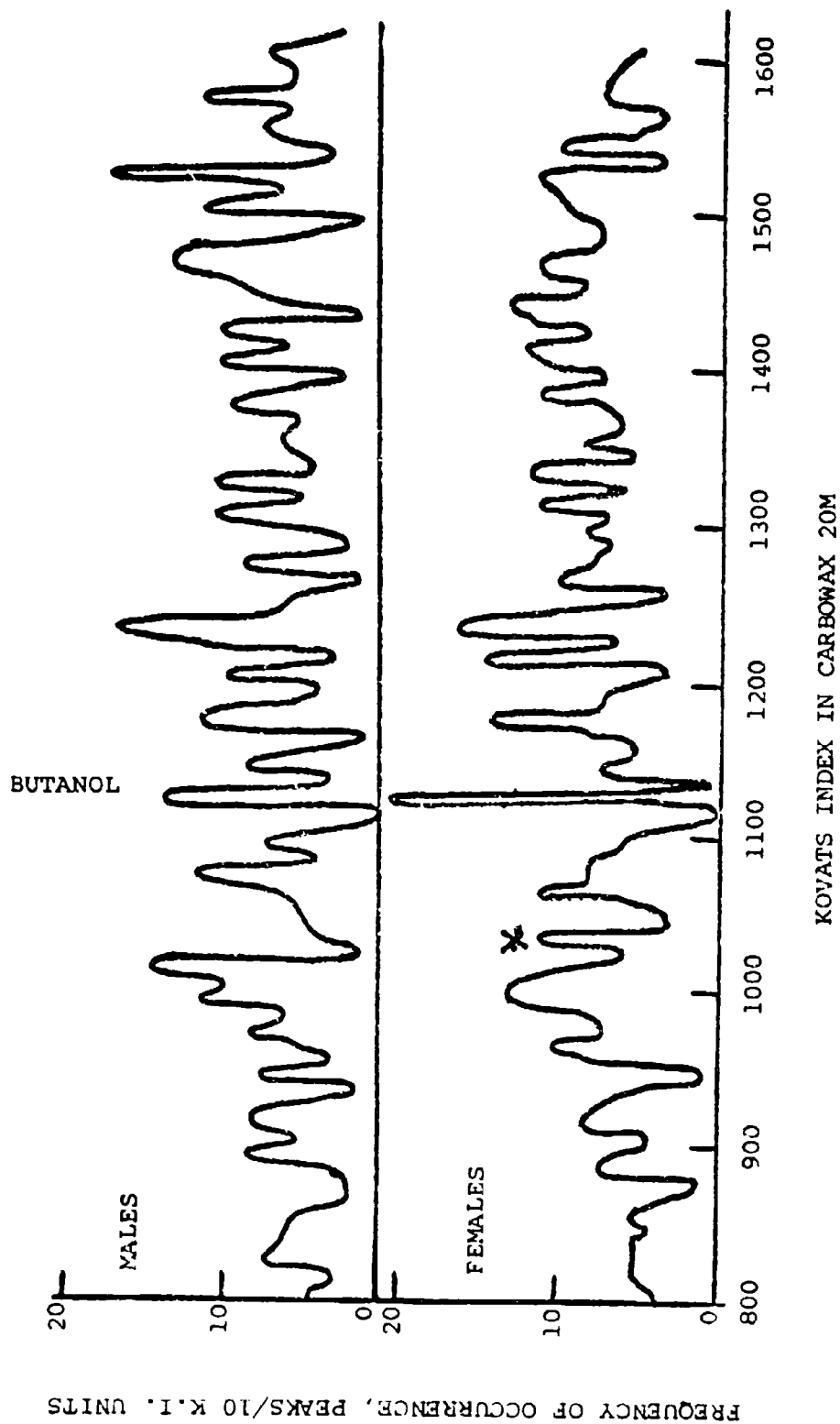


Figure 18
DENSITY OF OCCURRENCE OF PEAKS IN CHEMICAL SIGNATURES
OF CAUCASIAN MALES AND FEMALES ON A KOVATS INDEX PLOT

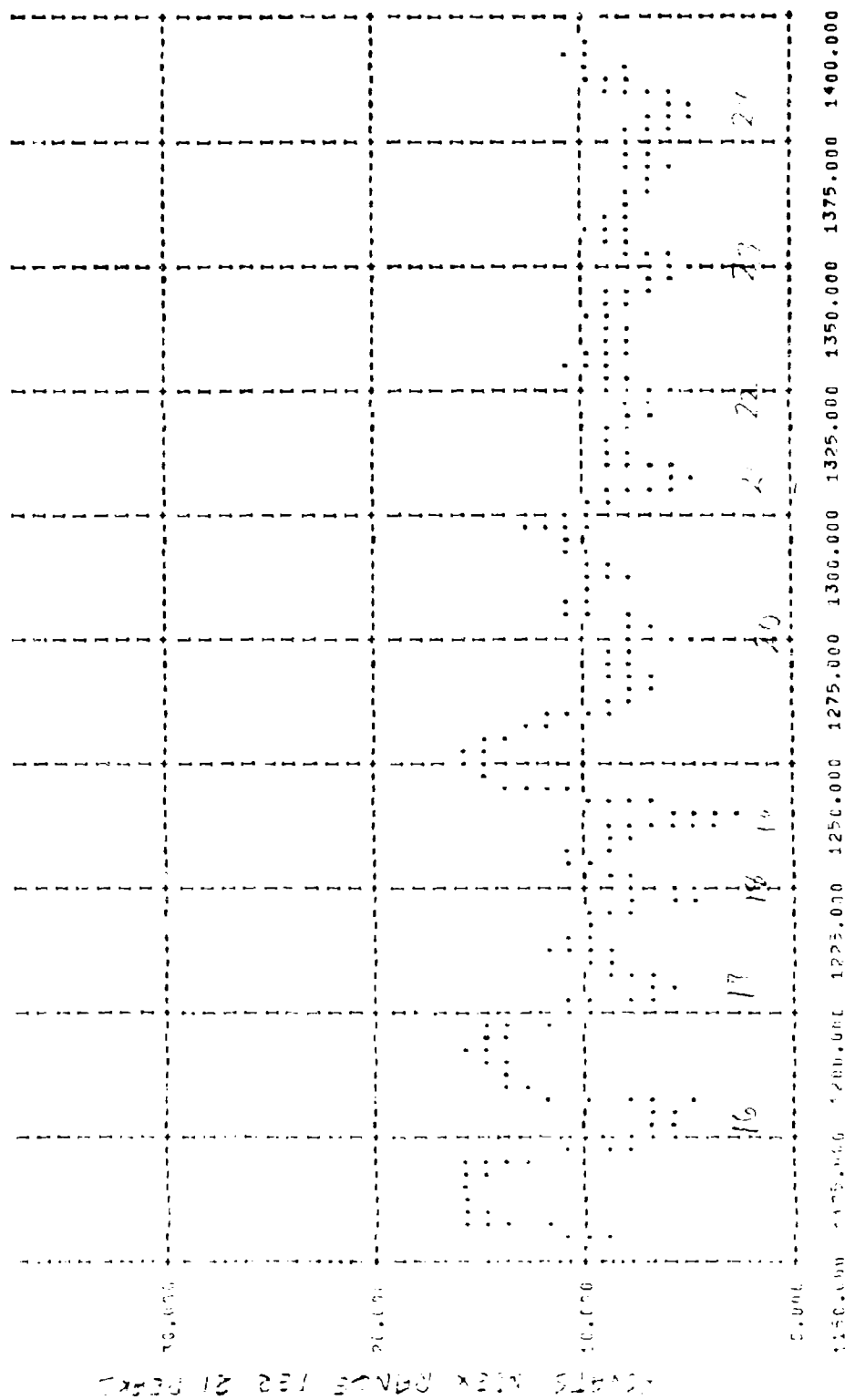


Figure 19
COMPUTER PRINTOUT OF HISTOGRAM OF PEAK OCCURRENCE
FREQUENCY IN KOVATS INDEX RANGE BETWEEN 1150 AND 1400 UNITS
IN POOLED CHROMATOGRAMS FROM ALL HUMANS STUDIED

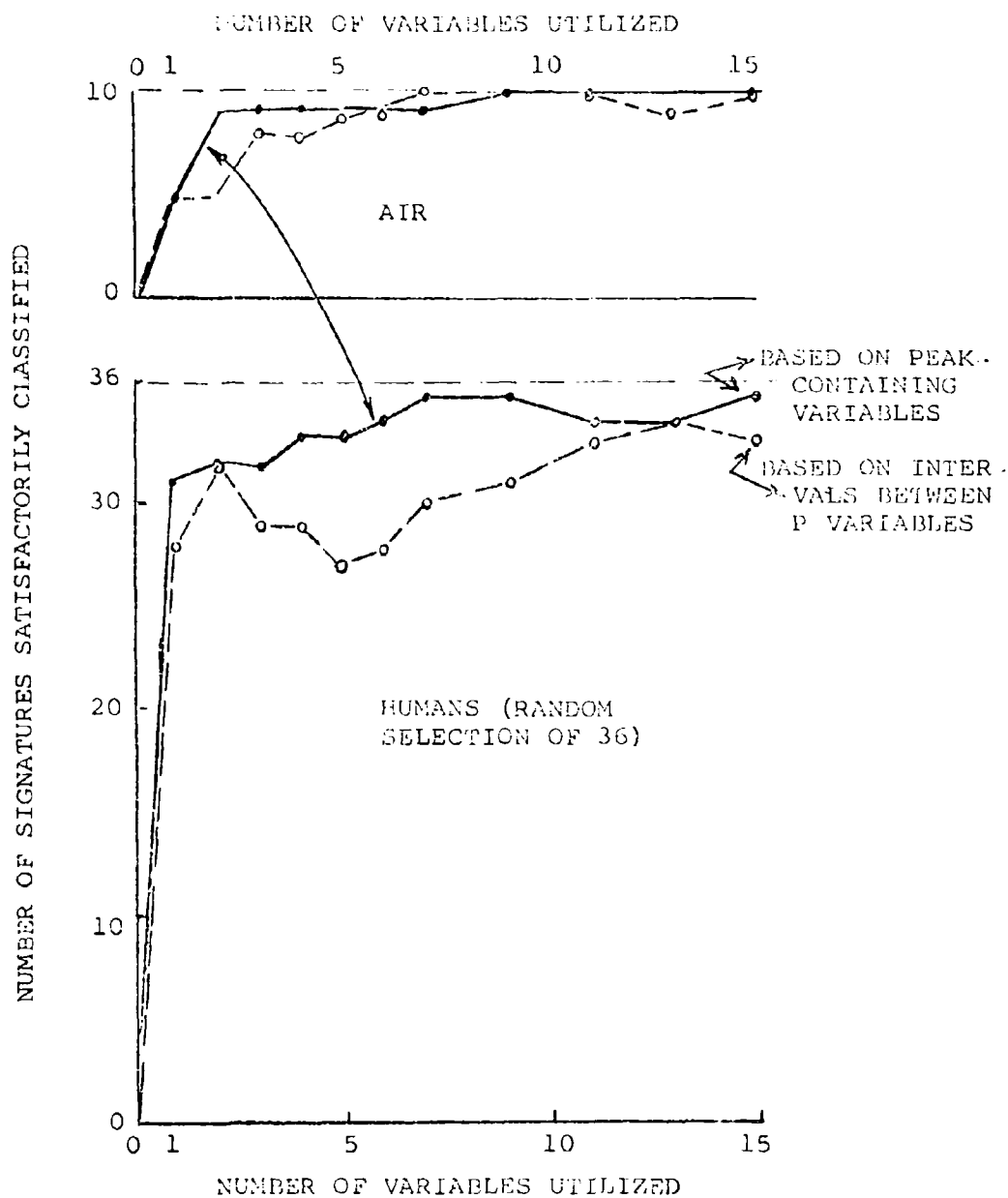


Figure 20
COMPARISON IN THE IMPROVEMENT OF TWO-WAY CLASSIFICATION
OF CHEMICAL SIGNATURES: USING VARIABLES (KOVATS INDEX RANGES)
THAT FREQUENTLY CONTAIN PEAKS IN HUMAN SIGNATURES
(P-VARIABLES, FULL LINE), OR VARIABLES THAT CORRESPOND TO
RANGES IN BETWEEN (DASHED LINE)

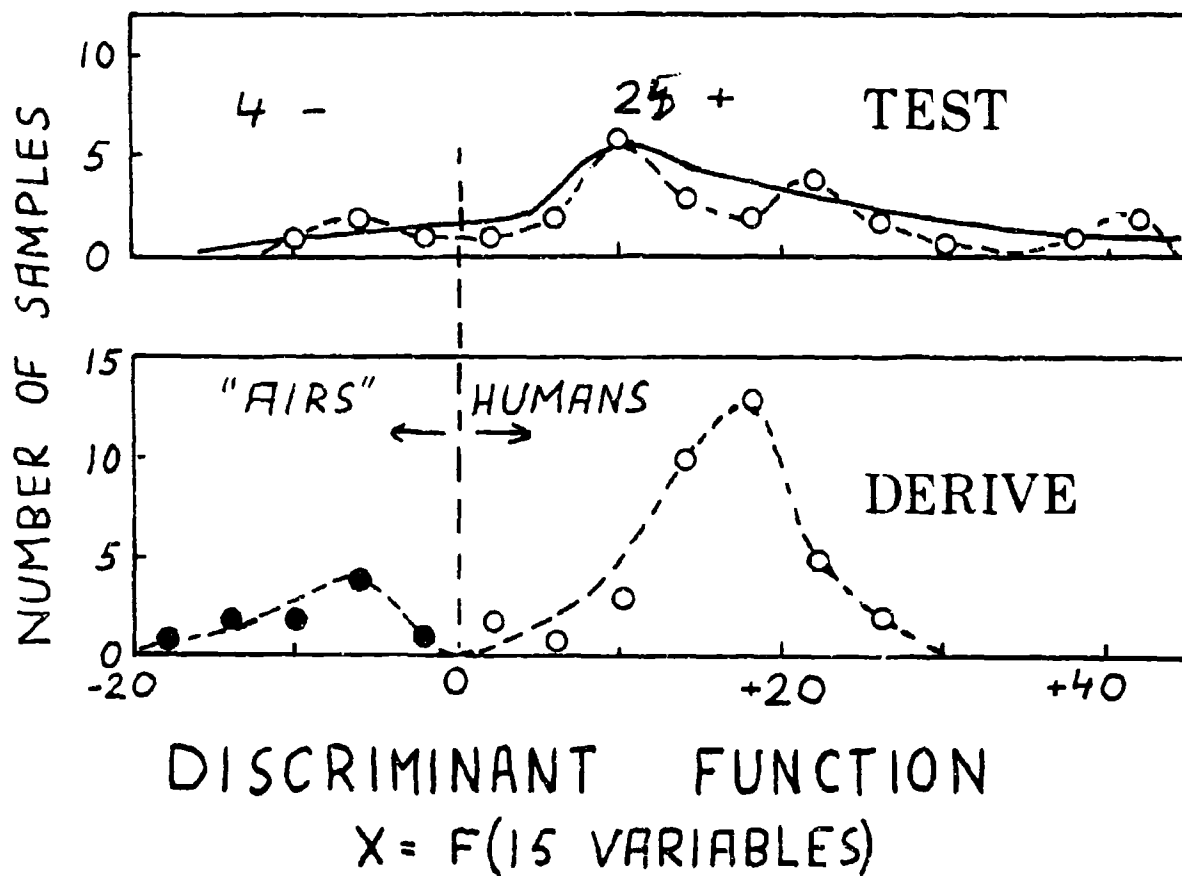


Figure 21

DERIVATION OF DISCRIMINANT FUNCTION FOR CLASSIFICATION
 OF HUMAN AND ENVIRONMENT CHEMICAL SIGNATURES
 AND TEST OF THE FUNCTION ON 29 ADDITIONAL HUMAN CHEMICAL SIGNATURES

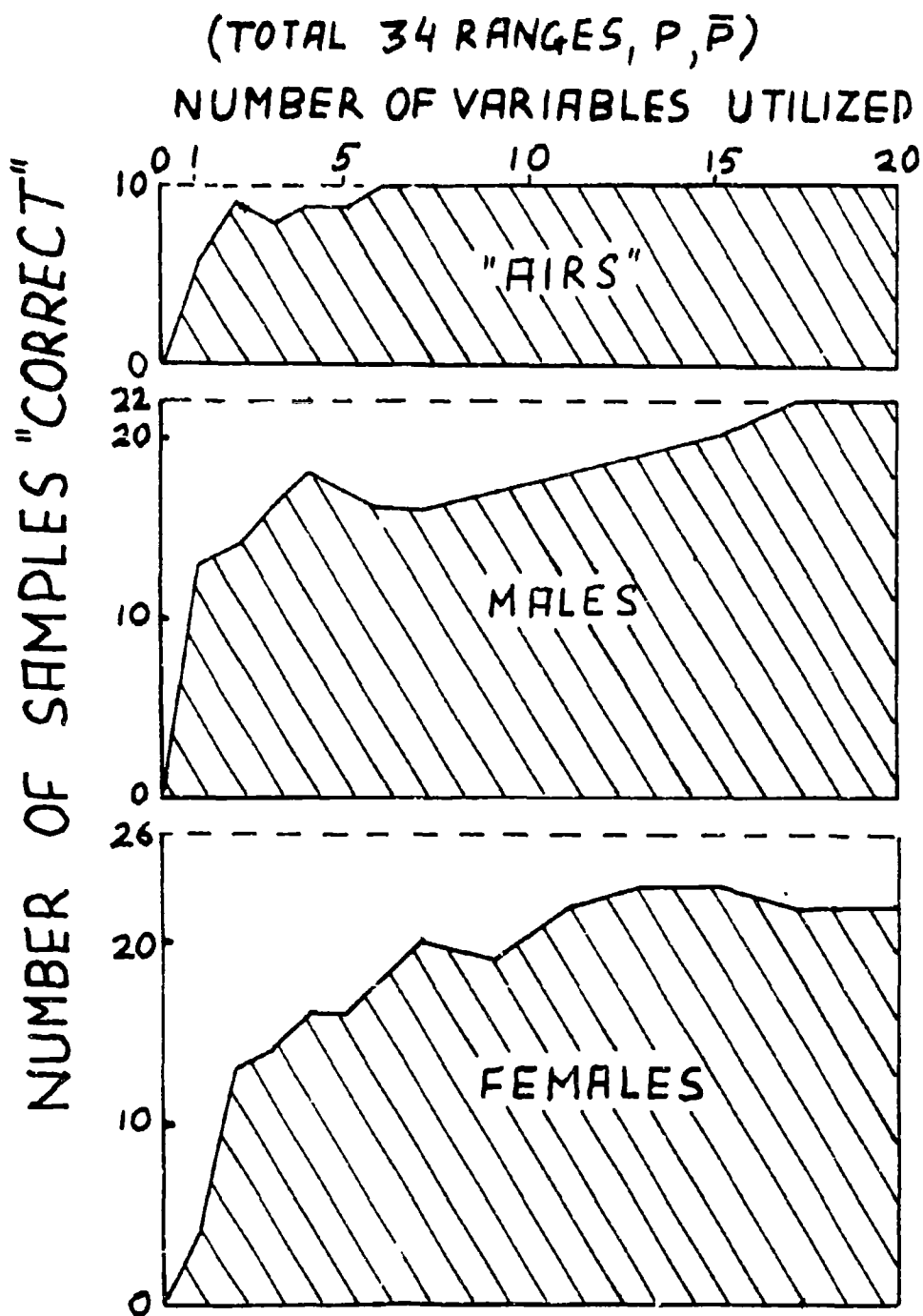


Figure 22
IMPROVEMENT IN CORRECT PATTERN ASSIGNMENTS WITH
INCREASE IN THE NUMBER OF VARIABLES UTILIZED BY THE
STEPWISE DISCRIMINANT ANALYSIS PROGRAM

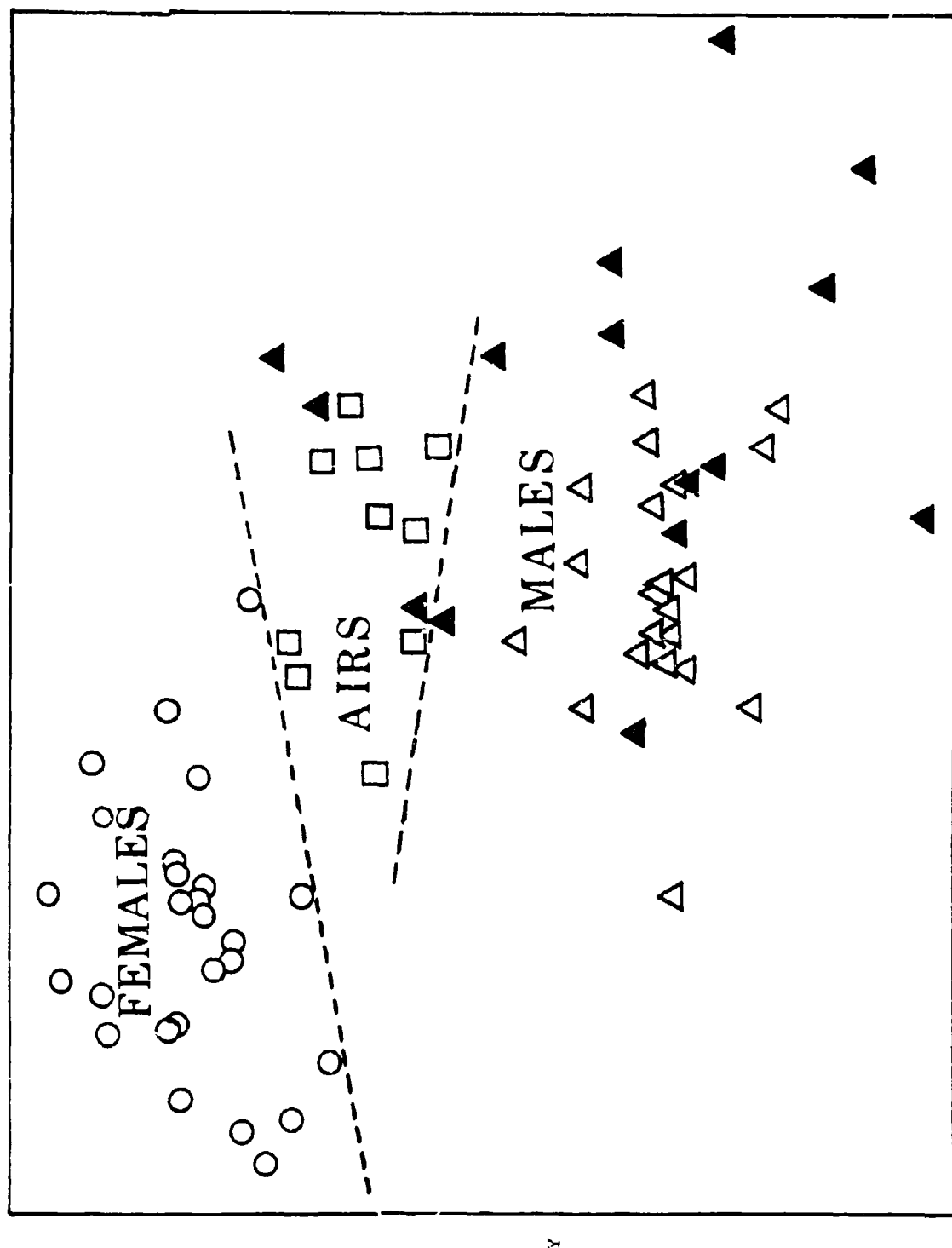


Figure 23
DERIVATION OF THREE-CLASS DISCRIMINANT FUNCTIONS FIELD
(HOLLOW SYMBOLS) AND TEST OF THE DERIVED FUNCTIONS WITH
ADDITIONAL MALE CHEMICAL SIGNATURE PATTERNS (FILLED TRIANGLES)

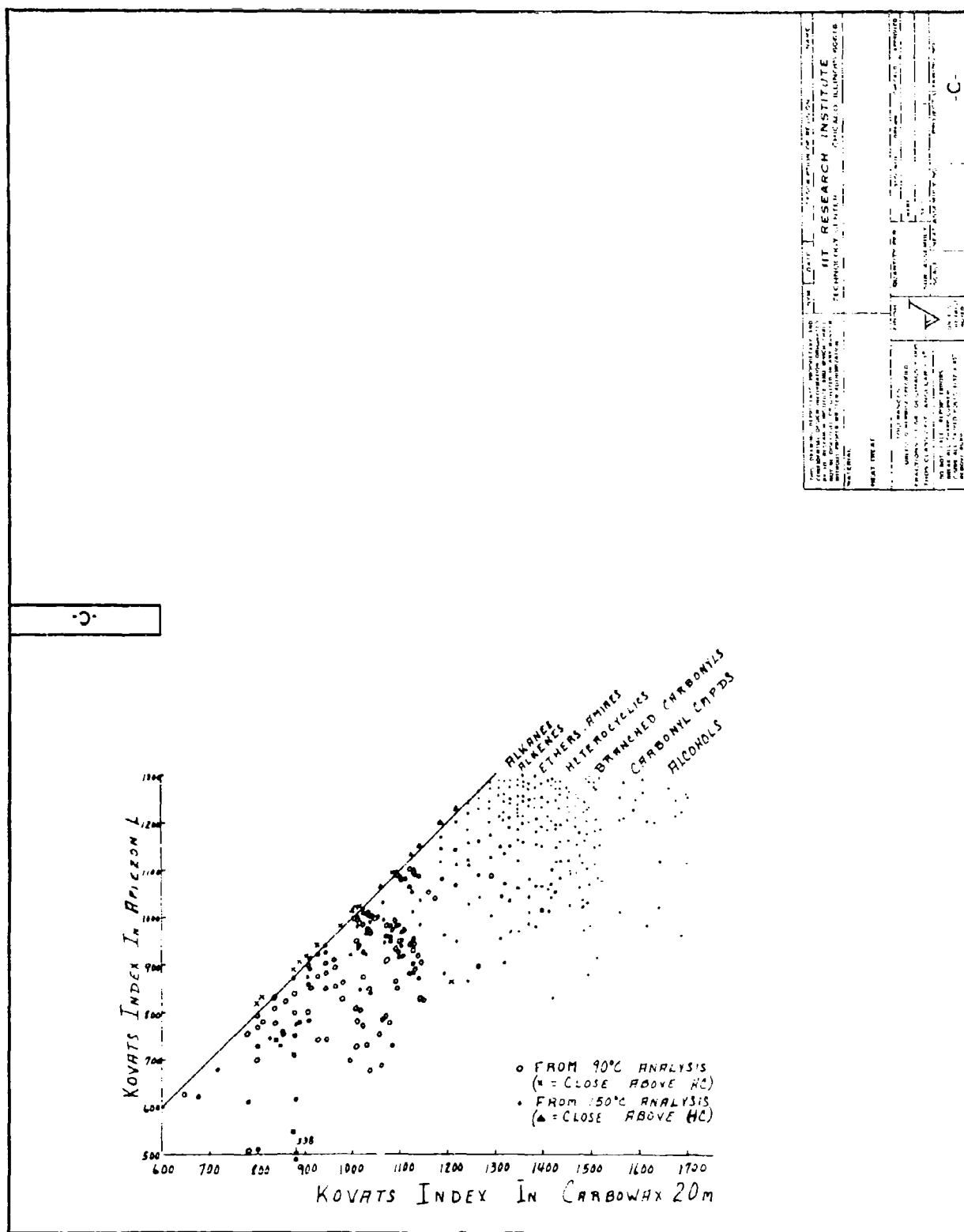


Figure 24

THREE-COLUMN DATA INTERPRETATION ASSISTED BY GA3COL
COMPUTER PROGRAM

SUBJECT: CAUCASIAN FEMALE L.B.

APPENDIX I
Tabulation of Chemical Signature Data

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EXPLANATION OF ARRANGEMENT

Human chemical signatures are arranged in numbered order, approximating the chronological order in which the data were calculated. The environment chemical signatures begin after the human chemical signature No. 69.

The tabulation is a copy of the computer printout.

The upper line in each pattern describes the source, ("SUBJECT"), gives the consecutive pattern number (one per pattern), and initials if the source was a human subject.

The second line is the data of the chemical signature collection.

The third line (Total Intensity) gives the total area, in sq. mm. at 0.1 x 1 electrometer setting, of all gas chromatographic peaks of the particular pattern, and the mean peak intensity (total area of the pattern's peaks divided by the number of peaks in the same pattern).

The columns in the tabulation are as follows:

- (1) First column- consecutive number of the peak in the particular pattern.
- (2) Second column - retention time in terms of Kovats retention index-cf. text, section 3.6.5.
- (3) Third column- the area of the peak, in sq. mm. calculated to the electrometer sensitivity setting of 0.1 x 1.
- (4) Fourth column- normalized area, equal to the area of the particular peak divided by the "Total Intensity"-- sum of areas of all peaks in the same pattern.

1

[illegible][illegible]

SUBJECT 10-1-64 XS

DATE 10-1-64
TOTAL INTENS 35316

PEAK	RETENTION TIME	AREA	NORMALIZED AREA	PEAK	RETENTION TIME	AREA	NORMALIZED AREA
1	524.96	532.	1.491	23	1217.76	210.	0.268
2	587.21	104.	0.282	24	1304.44	80.	1.074
3	632.20	303.	1.517	25	1319.02	3980.	4.995
4	727.66	3065.	8.274	26	1335.46	51.	0.643
5	815.62	815.	2.210	27	1358.19	50.	0.633
6	856.61	803.	2.167	28	1360.09	1008.	1.265
7	862.54	288.	0.790	29	1381.70	1776.	2.229
8	907.87	2565.	6.935	30	1398.39	560.	0.653
9	967.32	480.	1.350	31	1406.07	1632.	2.048
10	967.83	512.	1.387	32	1422.43	324.	4.076
11	987.23	7240.	6.065	33	1438.18	1120.	1.456
12	1007.88	480.	1.303	34	1452.39	1760.	2.209
13	1072.53	888.	2.495	35	1461.26	4592.	5.763
14	1121.91	720.	1.953	36	1488.28	448.	0.562
15	1162.53	832.	2.275	37	1507.45	5644.	7.063
16	1181.49	544.	1.474	38	1521.94	2280.	2.851
17	1202.04	32.	0.087	39	1541.48	1008.	1.265
18	1243.49	2015.	5.461	40	1547.60	112.	0.141
19	1244.71	1840.	4.984	41	1575.44	936.	1.175
				42	1597.44	2600.	3.263
				43	1625.57	3456.	4.337
				44	1642.14	960.	1.205

NOT REPRODUCIBLE

SUBJECT 10-1-64 XS

DATE 10-1-64
TOTAL INTENS 35316

PEAK	RETENTION TIME	AREA	NORMALIZED AREA	PEAK	RETENTION TIME	AREA	NORMALIZED AREA
1	524.96	532.	1.491	23	1217.76	210.	0.268
2	587.21	104.	0.282	24	1304.44	80.	1.074
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18	1243.49	2015.	5.461	40	1547.60	112.	0.141
19	1244.71	1840.	4.984	41	1575.44	936.	1.175
				42	1597.44	2600.	3.263
				43	1625.57	3456.	4.337
				44	1642.14	960.	1.205

NOT REPRODUCIBLE

DATE	DESCRIPTION	AMOUNT	BALANCE
10/1/74	10/1/74	100.00	100.00
10/2/74	10/2/74	100.00	200.00
10/3/74	10/3/74	100.00	300.00
10/4/74	10/4/74	100.00	400.00
10/5/74	10/5/74	100.00	500.00
10/6/74	10/6/74	100.00	600.00
10/7/74	10/7/74	100.00	700.00
10/8/74	10/8/74	100.00	800.00
10/9/74	10/9/74	100.00	900.00
10/10/74	10/10/74	100.00	1000.00
10/11/74	10/11/74	100.00	1100.00
10/12/74	10/12/74	100.00	1200.00
10/13/74	10/13/74	100.00	1300.00
10/14/74	10/14/74	100.00	1400.00
10/15/74	10/15/74	100.00	1500.00
10/16/74	10/16/74	100.00	1600.00
10/17/74	10/17/74	100.00	1700.00
10/18/74	10/18/74	100.00	1800.00
10/19/74	10/19/74	100.00	1900.00
10/20/74	10/20/74	100.00	2000.00

SUBJECT	INDR.	WLS	Bar	REAR. INTERACTIVE	REAR. INTERACTIVE
DATE	10-11-68	1958.			
TOTAL INTERVIEW					
PEAK	RETENTION TIME	FEES AREA	ADJUSTED AREA		
1	344.22	1.25	0.585		
2	400.55	4.25	2.397		
3	410.45	2.25	1.264		
4	728.72	2.25	1.264		
5	795.03	5.25	2.760		
6	842.18	4.25	2.395		
7	871.67	4.25	2.395		
8	907.65	17.25	9.806		
9	921.80	5.25	2.805		
10	957.15	7.25	3.747		
11	990.55	10.25	5.279		
12	1043.11	5.25	2.859		
13	1052.85	3.25	1.813		
14	1052.13	8.25	4.512		
15	1111.59	12.25	6.827		
16	1155.60	13.25	7.374		
17	1205.35	1.25	0.485		
18	1223.03	1.25	0.475		
19	1243.25	4.25	1.9271		

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[illegible]

SUBJECT	INSTR.	WAVE	SHA	WIRCHAMPT	453.	454.	455.
DATE	DATE	DATE	DATE	DATE	DATE	DATE	DATE
TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE	TOTAL INTENSIVE
1	525.56	432.	1.030	1.030	1.030	1.030	1.030
2	510.18	288.	0.667	0.667	0.667	0.667	0.667
3	654.74	354.	0.145	0.145	0.145	0.145	0.145
4	671.44	74.	2.052	2.052	2.052	2.052	2.052
5	742.77	693.	1.147	1.147	1.147	1.147	1.147
6	742.81	502.	2.384	2.384	2.384	2.384	2.384
7	822.67	1000.	0.725	0.725	0.725	0.725	0.725
8	872.98	304.	1.441	1.441	1.441	1.441	1.441
9	872.82	572.	1.441	1.441	1.441	1.441	1.441
10	915.12	520.	0.587	0.587	0.587	0.587	0.587
11	937.88	288.	0.667	0.667	0.667	0.667	0.667
12	966.77	483.	0.815	0.815	0.815	0.815	0.815
13	980.69	384.	2.803	2.803	2.803	2.803	2.803
14	994.95	1175.	2.803	2.803	2.803	2.803	2.803
15	1025.83	864.	2.803	2.803	2.803	2.803	2.803
16	1054.75	600.	0.677	0.677	0.677	0.677	0.677
17	1076.27	368.	0.994	0.994	0.994	0.994	0.994
18	1088.54	415.	2.817	2.817	2.817	2.817	2.817
19	1121.91	924.	1.448	1.448	1.448	1.448	1.448
20	1152.62	674.	0.444	0.444	0.444	0.444	0.444
21	1182.12	209.	1.441	1.441	1.441	1.441	1.441
22	1177.78	789.	1.441	1.441	1.441	1.441	1.441

SUBJECT 1017 JRP

DATE 11-11-66

TOTAL INTENSIVE 50244

PEAK	RETENTION TIME	AREA	PEAK AREA	NORMALIZED AREA
1	345.84	68	250	1.046
2	511.82	197	540	2.616
3	511.82	72	4000	16.348
4	511.82	66	480	1.902
5	742.04	572	120	0.490
6	742.04	200	1311.07	1.177
7	742.04	245	131.520	2.746
8	742.04	160	140	0.572
9	742.04	400	90	0.352
10	742.04	711.75	264	1.074
11	742.04	200	400	1.635
12	742.04	440	912	3.727
13	742.04	512	640	2.616
14	742.04	100.12	440	1.748
15	742.04	768	690	2.820
16	742.04	582	1452	5.934
17	742.04	1152	504	2.060
18	742.04	1004	196	0.801
19	742.04	1152.77	408	1.637
20	742.04	1052	1350	5.517

SUBJECT 1017 JRP

DATE 11-11-66

TOTAL INTENSIVE 50244

PEAK	RETENTION TIME	AREA	PEAK AREA	NORMALIZED AREA
1	345.84	68	250	1.046
2	511.82	197	540	2.616
3	511.82	72	4000	16.348
4	511.82	66	480	1.902
5	742.04	572	120	0.490
6	742.04	200	1311.07	1.177
7	742.04	245	131.520	2.746
8	742.04	160	140	0.572
9	742.04	400	90	0.352
10	742.04	711.75	264	1.074
11	742.04	200	400	1.635
12	742.04	440	912	3.727
13	742.04	512	640	2.616
14	742.04	100.12	440	1.748
15	742.04	768	690	2.820
16	742.04	582	1452	5.934
17	742.04	1152	504	2.060
18	742.04	1004	196	0.801
19	742.04	1152.77	408	1.637
20	742.04	1052	1350	5.517

NOT REPRODUCIBLE

SUBJECT				INDIAN MALE				MEH			
DATE				11-12-52				1-1-53			
TOTAL INTENSITY				14155				14155			
PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	52.47	100	0.337	1	1225.68	1360	0.337	1	1225.68	1360	0.337
2	52.47	100	0.337	2	1236.42	520	0.337	2	1236.42	520	0.337
3	52.47	100	0.337	3	1277.83	520	0.337	3	1277.83	520	0.337
4	52.47	100	0.337	4	1308.14	120	0.337	4	1308.14	120	0.337
5	52.47	100	0.337	5	1315.74	880	0.337	5	1315.74	880	0.337
6	52.47	100	0.337	6	1350.03	168	0.337	6	1350.03	168	0.337
7	52.47	100	0.337	7	1367.20	90	0.337	7	1367.20	90	0.337
8	52.47	100	0.337	8	1382.41	520	0.337	8	1382.41	520	0.337
9	52.47	100	0.337	9	1394.87	40	0.337	9	1394.87	40	0.337
10	52.47	100	0.337	10	1411.56	224	0.337	10	1411.56	224	0.337
11	52.47	100	0.337	11	1424.97	1296	0.337	11	1424.97	1296	0.337
12	52.47	100	0.337	12	1466.03	1040	0.337	12	1466.03	1040	0.337
13	52.47	100	0.337	13	1480.23	360	0.337	13	1480.23	360	0.337
14	52.47	100	0.337	14	1493.50	84	0.337	14	1493.50	84	0.337
15	52.47	100	0.337	15	1510.88	1840	0.337	15	1510.88	1840	0.337
16	52.47	100	0.337	16	1524.59	20	0.337	16	1524.59	20	0.337
17	52.47	100	0.337	17	1562.60	20	0.337	17	1562.60	20	0.337
18	52.47	100	0.337	18	1578.50	96	0.337	18	1578.50	96	0.337
19	52.47	100	0.337	19	1598.38	100	0.337	19	1598.38	100	0.337
20	52.47	100	0.337	20	1625.57	1440	0.337	20	1625.57	1440	0.337
21	52.47	100	0.337								

SUBJECT				INDIAN MALE				MEH			
DATE				11-12-52				1-1-53			
TOTAL INTENSITY				14155				14155			
PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	473.59	90	0.479	1	1225.68	1360	0.479	1	1225.68	1360	0.479
2	524.58	64	0.334	2	1236.42	520	0.334	2	1236.42	520	0.334
3	583.15	75	0.397	3	1277.83	520	0.397	3	1277.83	520	0.397
4	651.48	72	0.375	4	1308.14	120	0.375	4	1308.14	120	0.375
5	712.21	364	1.901	5	1315.74	880	1.901	5	1315.74	880	1.901
6	751.80	588	3.071	6	1350.03	168	3.071	6	1350.03	168	3.071
7	797.95	200	1.045	7	1367.20	90	1.045	7	1367.20	90	1.045
8	825.78	880	4.597	8	1382.41	520	4.597	8	1382.41	520	4.597
9	852.43	615	3.218	9	1394.87	40	3.218	9	1394.87	40	3.218
10	911.30	319	1.665	10	1411.56	224	1.665	10	1411.56	224	1.665
11	931.70	280	1.463	11	1424.97	1296	1.463	11	1424.97	1296	1.463
12	984.04	420	2.194	12	1466.03	1040	2.194	12	1466.03	1040	2.194
13	1002.22	392	2.048	13	1480.23	360	2.048	13	1480.23	360	2.048
14	1045.35	624	3.259	14	1493.50	84	3.259	14	1493.50	84	3.259
15	1052.50	476	2.486	15	1510.88	1840	2.486	15	1510.88	1840	2.486
16	1064.45	620	3.233	16	1524.59	20	3.233	16	1524.59	20	3.233
17	1093.16	368	1.922	17	1562.60	20	1.922	17	1562.60	20	1.922
18	1121.91	700	3.655	18	1578.50	96	3.655	18	1578.50	96	3.655
19	1163.66	480	2.507	19	1598.38	100	2.507	19	1598.38	100	2.507
20	1205.75	144	0.757	20	1625.57	1440	0.757	20	1625.57	1440	0.757

13

GVB

DATE

SUBJECT

TOTAL INTENSITY

DATE

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TOTAL INTENSITY

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TOTAL INTENSITY

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TOTAL INTENSITY

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SUBJECT

TOTAL INTENSITY

PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	561.97	2000	8.777	41	1224.33	735	2.070
2	565.97	720	2.003	42	1437.60	3520	9.874
3	569.10	4335	2.553	43	1287.82	364	1.021
4	573.19	984	2.760	44	1305.22	900	2.643
5	572.57	1832	4.578	45	1315.25	80	0.224
6	575.12	514	1.414	46	1345.93	280	0.765
7	575.93	672	1.085	47	1366.70	440	1.234
8	577.08	574	1.415	48	1380.54	308	0.864
9	578.24	400	1.345	49	1412.40	274	0.763
10	581.99	550	2.513	50	1418.10	190	0.550
11	585.99	413	2.269	51	1424.73	60	0.168
12	587.12	471	2.474	52	1457.33	720	2.020
13	588.42	1368	3.815	53	1479.25	610	1.726
14	588.77	164	0.471	54	1511.82	630	1.790
15	587.07	1200	3.265	55	1520.83	354	0.987
16	589.23	646	1.752	56	1534.89	570	1.534
17	591.91	1510	4.264	57	1554.73	150	0.421
18	595.06	1215	3.411	58	1580.02	30	0.084
19	596.24	1704	4.791	59	1588.92	280	0.802
20	597.57	1018	2.848	60	1600.67	308	0.864
				61	1625.57	2100	5.891

14

DM

DATE

SUBJECT

TOTAL INTENSITY

DATE

SUBJECT

TOTAL INTENSITY

DATE

SUBJECT

TOTAL INTENSITY

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SUBJECT

TOTAL INTENSITY

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SUBJECT

TOTAL INTENSITY

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SUBJECT

TOTAL INTENSITY

PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	579.95	400	0.845	20	1152.74	300	0.546
2	581.36	258	0.481	21	1176.01	210	0.374
3	582.27	1054	2.332	22	1203.54	1344	2.947
4	584.15	720	1.311	23	1234.95	1344	2.947
5	585.58	405	0.743	24	1249.70	1700	3.204
6	587.95	645	1.180	25	1274.71	756	1.376
7	589.75	1700	3.204	26	1301.37	1744	3.135
8	591.80	584	1.245	27	1324.88	1500	2.855
9	592.14	800	1.457	28	1371.06	480	0.824
10	594.34	918	1.661	29	1405.50	4500	8.284
11	597.82	1148	2.054	30	1441.57	1900	3.474
12	598.02	528	1.040	31	1456.80	800	1.457
13	601.28	528	0.961	32	1470.10	3000	5.462
14	602.54	124	0.233	33	1509.47	7100	12.954
15	604.73	532	0.969	34	1544.63	2052	3.730
16	607.13	552	1.005	35	1558.43	2800	5.244
17	609.06	174	0.320	36	1579.35	1664	3.050
18	609.15	532	0.965	37	1603.04	6765	12.317
19	611.97	750	1.375	38	1629.57	2810	5.127

1-8

SUBJECT WHITE MALE BB
 DATE 3-22-67
 TOTAL INTENSITY 1000

PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	9.00.00	3100	3.100	17	11.40.00	2250	2.250
2	9.00.00	4000	4.000	18	11.40.00	2730	2.730
3	11.00.00	2900	2.900	19	12.13.21	3200	3.200
4	10.00.00	1000	1.000	20	12.00.00	1320	1.320
5	10.00.00	1000	1.000	21	12.00.00	2170	2.170
6	10.00.00	1120	1.120	22	13.00.00	1530	1.530
7	10.00.00	1000	1.000	23	12.00.00	2200	2.200
8	10.00.00	1000	1.000	24	12.00.00	2200	2.200
9	10.00.00	1000	1.000	25	12.00.00	2200	2.200
10	10.00.00	1000	1.000	26	12.00.00	2200	2.200
11	10.00.00	1000	1.000	27	12.00.00	2200	2.200
12	10.00.00	1000	1.000	28	12.00.00	2200	2.200
13	10.00.00	1000	1.000	29	12.00.00	2200	2.200
14	10.00.00	1000	1.000	30	12.00.00	2200	2.200
15	10.00.00	1000	1.000	31	12.00.00	2200	2.200
16	10.00.00	1000	1.000	32	12.00.00	2200	2.200
				33	12.00.00	2200	2.200

SUBJECT WHITE MALE BB
 DATE 3-22-67
 TOTAL INTENSITY 1000

PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA	PEAK	RETENTION TIME	PEAK AREA	NORMALIZED AREA
1	9.00.00	3100	3.100	19	12.00.00	1000	1.000
2	9.00.00	4000	4.000	20	12.00.00	1000	1.000
3	11.00.00	2900	2.900	21	13.00.00	3670	3.670
4	10.00.00	1000	1.000	22	13.00.00	640	0.640
5	10.00.00	1000	1.000	23	13.00.00	2200	2.200
6	10.00.00	1000	1.000	24	13.00.00	330	0.330
7	10.00.00	1000	1.000	25	14.12.00	5580	5.580
8	10.00.00	1000	1.000	26	14.00.00	1320	1.320
9	10.00.00	1000	1.000	27	14.00.00	2000	2.000
10	10.00.00	1000	1.000	28	14.00.00	600	0.600
11	10.00.00	1000	1.000	29	15.12.00	4400	4.400
12	10.00.00	1000	1.000	30	15.00.00	1870	1.870
13	10.00.00	1000	1.000	31	15.00.00	640	0.640
14	10.00.00	1000	1.000	32	15.00.00	1190	1.190
15	10.00.00	1000	1.000	33	15.00.00	3900	3.900
16	10.00.00	1000	1.000	34	15.00.00	740	0.740
				35	15.00.00	8100	8.100
				36	15.00.00	2600	2.600

SUBJECT: WALTER H. ... SA
DATE: 3-21-61
TOTAL INTERVIEW: 1000.

20.41.2017 = 355.
11

PEAK	RETENTION TIME	AREA	PERCENT	AREA	PERCENT
1	4.74	100	100	100	100
2	5.14	100	100	100	100
3	5.54	100	100	100	100
4	7.17	100	100	100	100
5	8.14	100	100	100	100
6	9.14	100	100	100	100
7	9.54	100	100	100	100
8	9.94	100	100	100	100
9	10.34	100	100	100	100
10	10.74	100	100	100	100
11	11.14	100	100	100	100
12	11.54	100	100	100	100
13	11.94	100	100	100	100
14	12.34	100	100	100	100
15	12.74	100	100	100	100
16	13.14	100	100	100	100
17	13.54	100	100	100	100
18	13.94	100	100	100	100
19	14.34	100	100	100	100
20	14.74	100	100	100	100
21	15.14	100	100	100	100
22	15.54	100	100	100	100

SUBJECT DATE 3-22-67 INITIALS 100402

1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. 12. 13. 14. 15. 16. 17. 18. 19. 20. 21. 22. 23. 24. 25. 26. 27. 28. 29. 30. 31. 32. 33. 34. 35. 36. 37. 38. 39. 40. 41. 42. 43. 44. 45. 46. 47. 48. 49. 50. 51. 52. 53. 54. 55. 56. 57. 58. 59. 60. 61. 62. 63. 64. 65. 66. 67. 68. 69. 70. 71. 72. 73. 74. 75. 76. 77. 78. 79. 80. 81. 82. 83. 84. 85. 86. 87. 88. 89. 90. 91. 92. 93. 94. 95. 96. 97. 98. 99. 100. 101. 102. 103. 104. 105. 106. 107. 108. 109. 110. 111. 112. 113. 114. 115. 116. 117. 118. 119. 120. 121. 122. 123. 124. 125. 126. 127. 128. 129. 130. 131. 132. 133. 134. 135. 136. 137. 138. 139. 140. 141. 142. 143. 144. 145. 146. 147. 148. 149. 150. 151. 152. 153. 154. 155. 156. 157. 158. 159. 160. 161. 162. 163. 164. 165. 166. 167. 168. 169. 170. 171. 172. 173. 174. 175. 176. 177. 178. 179. 180. 181. 182. 183. 184. 185. 186. 187. 188. 189. 190. 191. 192. 193. 194. 195. 196. 197. 198. 199. 200. 201. 202. 203. 204. 205. 206. 207. 208. 209. 210. 211. 212. 213. 214. 215. 216. 217. 218. 219. 220. 221. 222. 223. 224. 225. 226. 227. 228. 229. 230. 231. 232. 233. 234. 235. 236. 237. 238. 239. 240. 241. 242. 243. 244. 245. 246. 247. 248. 249. 250. 251. 252. 253. 254. 255. 256. 257. 258. 259. 260. 261. 262. 263. 264. 265. 266. 267. 268. 269. 270. 271. 272. 273. 274. 275. 276. 277. 278. 279. 280. 281. 282. 283. 284. 285. 286. 287. 288. 289. 290. 291. 292. 293. 294. 295. 296. 297. 298. 299. 300. 301. 302. 303. 304. 305. 306. 307. 308. 309. 310. 311. 312. 313. 314. 315. 316. 317. 318. 319. 320. 321. 322. 323. 324. 325. 326. 327. 328. 329. 330. 331. 332. 333. 334. 335. 336. 337. 338. 339. 340. 341. 342. 343. 344. 345. 346. 347. 348. 349. 350. 351. 352. 353. 354. 355. 356. 357. 358. 359. 360. 361. 362. 363. 364. 365. 366. 367. 368. 369. 370. 371. 372. 373. 374. 375. 376. 377. 378. 379. 380. 381. 382. 383. 384. 385. 386. 387. 388. 389. 390. 391. 392. 393. 394. 395. 396. 397. 398. 399. 400. 401. 402. 403. 404. 405. 406. 407. 408. 409. 410. 411. 412. 413. 414. 415. 416. 417. 418. 419. 420. 421. 422. 423. 424. 425. 426. 427. 428. 429. 430. 431. 432. 433. 434. 435. 436. 437. 438. 439. 440. 441. 442. 443. 444. 445. 446. 447. 448. 449. 450. 451. 452. 453. 454. 455. 456. 457. 458. 459. 460. 461. 462. 463. 464. 465. 466. 467. 468. 469. 470. 471. 472. 473. 474. 475. 476. 477. 478. 479. 480. 481. 482. 483. 484. 485. 486. 487. 488. 489. 490. 491. 492. 493. 494. 495. 496. 497. 498. 499. 500. 501. 502. 503. 504. 505. 506. 507. 508. 509. 510. 511. 512. 513. 514. 515. 516. 517. 518. 519. 520. 521. 522. 523. 524. 525. 526. 527. 528. 529. 530. 531. 532. 533. 534. 535. 536. 537. 538. 539. 540. 541. 542. 543. 544. 545. 546. 547. 548. 549. 550. 551. 552. 553. 554. 555. 556. 557. 558. 559. 560. 561. 562. 563. 564. 565. 566. 567. 568. 569. 570. 571. 572. 573. 574. 575. 576. 577. 578. 579. 580. 581. 582. 583. 584. 585. 586. 587. 588. 589. 590. 591. 592. 593. 594. 595. 596. 597. 598. 599. 600. 601. 602. 603. 604. 605. 606. 607. 608. 609. 610. 611. 612. 613. 614. 615. 616. 617. 618. 619. 620. 621. 622. 623. 624. 625. 626. 627. 628. 629. 630. 631. 632. 633. 634. 635. 636. 637. 638. 639. 640. 641. 642. 643. 644. 645. 646. 647. 648. 649. 650. 651. 652. 653. 654. 655. 656. 657. 658. 659. 660. 661. 662. 663. 664. 665. 666. 667. 668. 669. 670. 671. 672. 673. 674. 675. 676. 677. 678. 679. 680. 681. 682. 683. 684. 685. 686. 687. 688. 689. 690. 691. 692. 693. 694. 695. 696. 697. 698. 699. 700. 701. 702. 703. 704. 705. 706. 707. 708. 709. 710. 711. 712. 713. 714. 715. 716. 717. 718. 719. 720. 721. 722. 723. 724. 725. 726. 727. 728. 729. 730. 731. 732. 733. 734. 735. 736. 737. 738. 739. 740. 741. 742. 743. 744. 745. 746. 747. 748. 749. 750. 751. 752. 753. 754. 755. 756. 757. 758. 759. 760. 761. 762. 763. 764. 765. 766. 767. 768. 769. 770. 771. 772. 773. 774. 775. 776. 777. 778. 779. 780. 781. 782. 783. 784. 785. 786. 787. 788. 789. 790. 791. 792. 793. 794. 795. 796. 797. 798. 799. 800. 801. 802. 803. 804. 805. 806. 807. 808. 809. 810. 811. 812. 813. 814. 815. 816. 817. 818. 819. 820. 821. 822. 823. 824. 825. 826. 827. 828. 829. 830. 831. 832. 833. 834. 835. 836. 837. 838. 839. 840. 84

| PEAK | RETENTION TIME | WAVE LENGTH | WAVELENGTH AREA | WAVELENGTH |
|------|----------------|-------------|-----------------|------------|
| 1 | 52.54 | 4250 | 1000 | 1000 |
| 2 | 57.56 | 4250 | 975 | 1000 |
| 3 | 75.52 | 4250 | 1000 | 1000 |
| 4 | 78.50 | 4250 | 1000 | 1000 |
| 5 | 80.50 | 4250 | 1000 | 1000 |
| 6 | 81.50 | 4250 | 1000 | 1000 |
| 7 | 82.50 | 4250 | 1000 | 1000 |
| 8 | 83.50 | 4250 | 1000 | 1000 |
| 9 | 84.50 | 4250 | 1000 | 1000 |
| 10 | 85.50 | 4250 | 1000 | 1000 |
| 11 | 86.50 | 4250 | 1000 | 1000 |
| 12 | 87.50 | 4250 | 1000 | 1000 |
| 13 | 88.50 | 4250 | 1000 | 1000 |
| 14 | 89.50 | 4250 | 1000 | 1000 |
| 15 | 90.50 | 4250 | 1000 | 1000 |
| 16 | 91.50 | 4250 | 1000 | 1000 |
| 17 | 92.50 | 4250 | 1000 | 1000 |
| 18 | 93.50 | 4250 | 1000 | 1000 |

NOT REPRODUCIBLE

| SUBJECT | DATE | WAVELENGTH | GE | 17 | 18 | 19 | 20 |
|-----------------|----------------|------------|----------------|-----------|----------------|-----------|----------------|
| DATE | 3-25-67 | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 |
| TOTAL INTENSITY | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 |
| PEAK | RETENTION TIME | PEAK AREA | RETENTION TIME | PEAK AREA | RETENTION TIME | PEAK AREA | RETENTION TIME |
| 1 | 407.23 | 1368 | 14 | 1742 | 1445.15 | 5019 | 1445.15 |
| 2 | 524.96 | 3363 | 19 | 5440 | 1193.18 | 3831 | 1193.18 |
| 3 | 524.96 | 3363 | 20 | 9440 | 1233.04 | 6547 | 1233.04 |
| 4 | 751.02 | 543 | 21 | 3608 | 1304.19 | 4861 | 1304.19 |
| 5 | 792.77 | 384 | 22 | 200 | 1337.57 | 0141 | 1337.57 |
| 6 | 815.62 | 444 | 23 | 2308 | 1356.13 | 1822 | 1356.13 |
| 7 | 647.97 | 1908 | 24 | 330 | 1373.18 | 0237 | 1373.18 |
| 8 | 875.25 | 768 | 25 | 8448 | 1406.83 | 5949 | 1406.83 |
| 9 | 914.09 | 768 | 26 | 2592 | 1441.83 | 2817 | 1441.83 |
| 10 | 975.23 | 2448 | 27 | 1680 | 1460.35 | 1833 | 1460.35 |
| 11 | 995.41 | 4952 | 28 | 1680 | 1470.89 | 1833 | 1470.89 |
| 12 | 1013.85 | 4874 | 29 | 13272 | 1508.51 | 9365 | 1508.51 |
| 13 | 1084.54 | 6400 | 30 | 15188 | 1518.84 | 2293 | 1518.84 |
| 14 | 1099.65 | 1944 | 31 | 4597 | 1547.05 | 1352 | 1547.05 |
| 15 | 1121.81 | 1944 | 32 | 624 | 1556.46 | 3707 | 1556.46 |
| 16 | 1159.29 | 1083 | 33 | 3920 | 1577.35 | 0856 | 1577.35 |
| 17 | 1175.23 | 1609 | 34 | 572 | 1609.24 | 8762 | 1609.24 |
| 18 | 1195.25 | 384 | 35 | 1244 | 1625.57 | 3002 | 1625.57 |
| 19 | 1225.54 | 3996 | | 404 | | | |
| 20 | 1245.44 | 1040 | | | | | |

| SUBJECT | DATE | WAVELENGTH | TP | 20 | 21 | 22 | 23 |
|-----------------|----------------|------------|----------------|-----------|----------------|-----------|----------------|
| DATE | 3-25-67 | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 |
| TOTAL INTENSITY | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 | 127942 |
| PEAK | RETENTION TIME | PEAK AREA | RETENTION TIME | PEAK AREA | RETENTION TIME | PEAK AREA | RETENTION TIME |
| 1 | 407.23 | 1368 | 21 | 1742 | 1256.13 | 1401 | 1256.13 |
| 2 | 524.96 | 3363 | 22 | 2320 | 1271.74 | 1813 | 1271.74 |
| 3 | 524.96 | 3363 | 23 | 280 | 1308.39 | 0219 | 1308.39 |
| 4 | 751.02 | 543 | 24 | 3296 | 1327.42 | 2576 | 1327.42 |
| 5 | 792.77 | 384 | 25 | 180 | 1359.27 | 0125 | 1359.27 |
| 6 | 815.62 | 444 | 26 | 2088 | 1370.57 | 1632 | 1370.57 |
| 7 | 647.97 | 1908 | 27 | 312 | 1392.52 | 0244 | 1392.52 |
| 8 | 875.25 | 768 | 28 | 80 | 1408.03 | 0063 | 1408.03 |
| 9 | 914.09 | 768 | 29 | 6888 | 1427.14 | 5386 | 1427.14 |
| 10 | 975.23 | 2448 | 30 | 2448 | 1461.87 | 1913 | 1461.87 |
| 11 | 995.41 | 4952 | 31 | 1232 | 1479.04 | 0963 | 1479.04 |
| 12 | 1013.85 | 4874 | 32 | 630 | 1491.08 | 0649 | 1491.08 |
| 13 | 1084.54 | 6400 | 33 | 192 | 1500.50 | 0150 | 1500.50 |
| 14 | 1099.65 | 1944 | 34 | 12100 | 1526.77 | 9504 | 1526.77 |
| 15 | 1121.81 | 1944 | 35 | 3600 | 1534.98 | 2814 | 1534.98 |
| 16 | 1159.29 | 1083 | 36 | 2520 | 1564.24 | 1570 | 1564.24 |
| 17 | 1175.23 | 1609 | 37 | 3584 | 1574.94 | 2801 | 1574.94 |
| 18 | 1195.25 | 384 | 38 | 1428 | 1593.94 | 1116 | 1593.94 |
| 19 | 1225.54 | 3996 | 39 | 15232 | 1625.57 | 11903 | 1625.57 |
| 20 | 1245.44 | 1040 | 40 | 4536 | 1641.83 | 3545 | 1641.83 |

SUBJECT WHITE MALE RR 21
 DATE 3-30-57
 TOTAL INTENSIVE 134700
 PEAK INTENSIVE 3745

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 50.15 | 3070 | 4.37 | 19 | 1252.99 | 12050 | 9.378 |
| 2 | 57.50 | 1030 | 1.43 | 20 | 1275.40 | 384 | 0.285 |
| 3 | 74.53 | 705 | 0.97 | 21 | 1324.23 | 4000 | 3.557 |
| 4 | 79.57 | 805 | 0.86 | 22 | 1357.74 | 330 | 0.249 |
| 5 | 84.34 | 410 | 0.50 | 23 | 1374.17 | 2500 | 1.897 |
| 6 | 85.42 | 315 | 0.38 | 24 | 1397.17 | 240 | 0.176 |
| 7 | 86.91 | 1605 | 1.87 | 25 | 1424.65 | 7200 | 5.394 |
| 8 | 91.94 | 1130 | 1.35 | 26 | 1460.32 | 2800 | 2.134 |
| 9 | 95.17 | 950 | 0.95 | 27 | 1475.38 | 2400 | 1.808 |
| 10 | 97.55 | 605 | 0.64 | 28 | 1493.35 | 700 | 0.522 |
| 11 | 104.08 | 170 | 0.20 | 29 | 1502.82 | 330 | 0.249 |
| 12 | 105.53 | 1870 | 1.85 | 30 | 1525.41 | 13450 | 9.953 |
| 13 | 107.59 | 430 | 0.50 | 31 | 1534.63 | 260 | 1.947 |
| 14 | 112.01 | 445 | 0.51 | 32 | 1564.32 | 2570 | 1.909 |
| 15 | 113.41 | 4750 | 4.75 | 33 | 1576.01 | 3300 | 2.450 |
| 16 | 1175.35 | 4400 | 3.24 | 34 | 1592.42 | 1500 | 1.174 |
| 17 | 1187.79 | 2900 | 2.90 | 35 | 1625.57 | 14500 | 10.780 |
| 18 | 1200.52 | 5350 | 5.35 | 36 | 1641.61 | 5100 | 3.951 |

SUBJECT WHITE MALE KW 22
 DATE 3-30-57
 TOTAL INTENSIVE 87550
 PEAK INTENSIVE 4407

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 47.24 | 2490 | 0.309 | 17 | 1276.57 | 3740 | 4.673 |
| 2 | 55.51 | 530 | 0.65 | 18 | 1305.71 | 5250 | 6.504 |
| 3 | 74.41 | 3440 | 4.19 | 19 | 1325.25 | 2350 | 2.914 |
| 4 | 80.12 | 250 | 0.31 | 20 | 1336.93 | 21700 | 2.695 |
| 5 | 83.33 | 4500 | 5.51 | 21 | 1368.61 | 36400 | 4.507 |
| 6 | 85.40 | 3770 | 4.64 | 22 | 1422.01 | 3500 | 4.339 |
| 7 | 85.67 | 480 | 0.59 | 23 | 1440.70 | 7400 | 9.20 |
| 8 | 97.33 | 1285 | 1.58 | 24 | 1458.77 | 12700 | 1.575 |
| 9 | 99.59 | 3040 | 3.72 | 25 | 1485.34 | 15300 | 1.886 |
| 10 | 105.89 | 2300 | 2.83 | 26 | 1505.50 | 890 | 0.111 |
| 11 | 111.91 | 4100 | 5.04 | 27 | 1525.94 | 11000 | 1.367 |
| 12 | 115.03 | 4800 | 5.94 | 28 | 1531.43 | 9210 | 1.141 |
| 13 | 1175.49 | 3700 | 4.60 | 29 | 1561.82 | 3300 | 0.410 |
| 14 | 1205.34 | 2690 | 3.32 | 30 | 1574.07 | 3450 | 0.428 |
| 15 | 1221.07 | 5040 | 6.24 | 31 | 1592.52 | 1470 | 0.180 |
| 16 | 1231.41 | 4000 | 4.95 | 32 | 1625.57 | 9700 | 1.233 |
| | | | | 33 | 1642.88 | 2370 | 0.294 |

SUBJECT MALAYAN
DATE 4-5-67
TOTAL INTENSITY 11325.0

25

| PEAK | RETENTION TIME | AREA | PEAK AREA | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|------|-----------|----------------|-----------|-----------------|
| 1 | 475.00 | 400 | 400 | 17 | 1644.91 | 2.350 |
| 2 | 527.75 | 300 | 300 | 18 | 1774.0 | 2.524 |
| 3 | 555.45 | 300 | 300 | 19 | 1800.0 | 2.560 |
| 4 | 735.00 | 700 | 700 | 20 | 1839.31 | 2.620 |
| 5 | 765.00 | 700 | 700 | 21 | 1855.02 | 2.650 |
| 6 | 805.00 | 700 | 700 | 22 | 1875.71 | 2.680 |
| 7 | 825.00 | 700 | 700 | 23 | 1880.00 | 2.690 |
| 8 | 835.00 | 700 | 700 | 24 | 1885.73 | 2.700 |
| 9 | 905.00 | 700 | 700 | 25 | 1885.17 | 2.700 |
| 10 | 925.00 | 700 | 700 | 26 | 1885.05 | 2.700 |
| 11 | 955.00 | 700 | 700 | 27 | 1885.05 | 2.700 |
| 12 | 965.00 | 700 | 700 | 28 | 1885.05 | 2.700 |
| 13 | 1005.00 | 700 | 700 | 29 | 1885.05 | 2.700 |
| 14 | 1055.00 | 700 | 700 | 30 | 1885.05 | 2.700 |
| 15 | 1105.00 | 700 | 700 | 31 | 1885.05 | 2.700 |
| 16 | 1155.00 | 700 | 700 | 32 | 1885.05 | 2.700 |
| 17 | 1175.00 | 700 | 700 | 33 | 1885.05 | 2.700 |

SUBJECT WHITE
DATE 4-5-67
TOTAL INTENSITY 11325.0

26

| PEAK | RETENTION TIME | AREA | PEAK AREA | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|------|-----------|----------------|-----------|-----------------|
| 1 | 527.75 | 400 | 400 | 20 | 1885.05 | 2.700 |
| 2 | 600.00 | 400 | 400 | 21 | 1885.05 | 2.700 |
| 3 | 625.00 | 400 | 400 | 22 | 1885.05 | 2.700 |
| 4 | 675.00 | 400 | 400 | 23 | 1885.05 | 2.700 |
| 5 | 735.00 | 400 | 400 | 24 | 1885.05 | 2.700 |
| 6 | 765.00 | 400 | 400 | 25 | 1885.05 | 2.700 |
| 7 | 805.00 | 400 | 400 | 26 | 1885.05 | 2.700 |
| 8 | 825.00 | 400 | 400 | 27 | 1885.05 | 2.700 |
| 9 | 835.00 | 400 | 400 | 28 | 1885.05 | 2.700 |
| 10 | 905.00 | 400 | 400 | 29 | 1885.05 | 2.700 |
| 11 | 925.00 | 400 | 400 | 30 | 1885.05 | 2.700 |
| 12 | 955.00 | 400 | 400 | 31 | 1885.05 | 2.700 |
| 13 | 1005.00 | 400 | 400 | 32 | 1885.05 | 2.700 |
| 14 | 1055.00 | 400 | 400 | 33 | 1885.05 | 2.700 |
| 15 | 1105.00 | 400 | 400 | 34 | 1885.05 | 2.700 |
| 16 | 1155.00 | 400 | 400 | 35 | 1885.05 | 2.700 |
| 17 | 1175.00 | 400 | 400 | 36 | 1885.05 | 2.700 |
| 18 | 1185.00 | 400 | 400 | 37 | 1885.05 | 2.700 |
| 19 | 1195.00 | 400 | 400 | 38 | 1885.05 | 2.700 |

SUBJECT: WHITELAND LON
DATE: 3-15-67
TOTAL INTENSITY: 150000

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 475.92 | 3.34 | 6.346 | 20 | 1170.53 | 3260 | 2.524 |
| 2 | 513.17 | 1.00 | 1.936 | 21 | 1203.54 | 4362 | 3.405 |
| 3 | 653.61 | 351 | 6.712 | 22 | 1259.05 | 4410 | 3.434 |
| 4 | 700.27 | 1.00 | 1.936 | 23 | 1275.19 | 2010 | 1.531 |
| 5 | 773.08 | 1175 | 2.243 | 24 | 1306.01 | 4810 | 3.858 |
| 6 | 804.57 | 1440 | 1.074 | 25 | 1317.37 | 2640 | 2.065 |
| 7 | 849.19 | 4949 | 3.163 | 26 | 1340.29 | 1344 | 1.041 |
| 8 | 850.82 | 1472 | 3.406 | 27 | 1361.01 | 4800 | 3.851 |
| 9 | 864.42 | 1450 | 1.272 | 28 | 1379.17 | 1960 | 1.545 |
| 10 | 901.59 | 3635 | 2.574 | 29 | 1415.36 | 8950 | 7.055 |
| 11 | 903.73 | 4081 | 3.105 | 30 | 1452.41 | 3674 | 2.879 |
| 12 | 945.14 | 472 | 3.402 | 31 | 1470.05 | 2470 | 1.976 |
| 13 | 1028.59 | 144 | 1.407 | 32 | 1479.80 | 3744 | 2.944 |
| 14 | 1074.57 | 234 | 2.405 | 33 | 1498.07 | 1344 | 1.041 |
| 15 | 1202.59 | 6910 | 2.203 | 34 | 1523.56 | 12604 | 9.873 |
| 16 | 1051.04 | 4400 | 3.401 | 35 | 1561.45 | 2510 | 1.956 |
| 17 | 1097.97 | 3720 | 2.474 | 36 | 1574.13 | 2800 | 2.127 |
| 18 | 1121.91 | 7600 | 1.523 | 37 | 1589.66 | 4600 | 3.629 |
| 19 | 1155.21 | 5512 | 3.427 | 38 | 1625.57 | 10440 | 7.924 |
| | | | | 39 | 1643.53 | 1630 | 1.244 |

28

SUBJECT: WHITELAND VR
DATE: 3-15-67
TOTAL INTENSITY: 85730

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 525.70 | 5815 | 7.950 | 18 | 1230.40 | 6680 | 7.791 |
| 2 | 675.30 | 1120 | 1.304 | 19 | 1241.98 | 4750 | 5.552 |
| 3 | 747.41 | 4824 | 3.423 | 20 | 1262.11 | 1824 | 2.126 |
| 4 | 811.43 | 1524 | 1.177 | 21 | 1312.03 | 900 | 1.020 |
| 5 | 834.33 | 4784 | 3.427 | 22 | 1318.96 | 974 | 1.134 |
| 6 | 844.57 | 1664 | 1.241 | 23 | 1339.45 | 274 | 0.317 |
| 7 | 913.37 | 4220 | 3.172 | 24 | 1356.48 | 390 | 0.450 |
| 8 | 953.05 | 1472 | 1.177 | 25 | 1376.75 | 352 | 0.411 |
| 9 | 981.18 | 10128 | 1.841 | 26 | 1394.25 | 864 | 1.008 |
| 10 | 1032.08 | 2680 | 3.175 | 27 | 1425.60 | 1520 | 1.773 |
| 11 | 1056.62 | 1100 | 1.283 | 28 | 1447.95 | 264 | 0.308 |
| 12 | 1084.79 | 5064 | 5.906 | 29 | 1472.91 | 2360 | 2.760 |
| 13 | 1105.82 | 800 | 0.933 | 30 | 1519.22 | 720 | 0.840 |
| 14 | 1121.91 | 2520 | 2.934 | 31 | 1530.38 | 1070 | 1.254 |
| 15 | 1122.58 | 1424 | 1.625 | 32 | 1548.13 | 1260 | 1.470 |
| 16 | 1152.13 | 5184 | 6.046 | 33 | 1564.00 | 472 | 0.517 |
| 17 | 1211.75 | 1220 | 1.423 | 34 | 1625.57 | 880 | 1.036 |
| | | | | 35 | 1641.59 | 510 | 0.595 |

| SUBJECT | DATE | TIME | SR | RC | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK AREA | NORMALIZED AREA |
|---------|----------|-------|------|----|----------------|-----------|-----------------|-----------|-----------------|
| 1 | 12/10/67 | 14:00 | 2580 | 19 | 1240.24 | 27.00 | 2.845 | 1240.24 | 27.00 |
| 2 | 12/10/67 | 14:00 | 2580 | 20 | 1260.20 | 760 | 0.795 | 1260.20 | 760 |
| 3 | 12/10/67 | 14:00 | 2580 | 21 | 1272.54 | 480 | 0.341 | 1272.54 | 480 |
| 4 | 12/10/67 | 14:00 | 2580 | 22 | 1294.80 | 950 | 0.870 | 1294.80 | 950 |
| 5 | 12/10/67 | 14:00 | 2580 | 23 | 1304.30 | 3750 | 3.925 | 1304.30 | 3750 |
| 6 | 12/10/67 | 14:00 | 2580 | 24 | 1358.56 | 780 | 0.793 | 1358.56 | 780 |
| 7 | 12/10/67 | 14:00 | 2580 | 25 | 1379.06 | 2080 | 2.170 | 1379.06 | 2080 |
| 8 | 12/10/67 | 14:00 | 2580 | 26 | 1394.73 | 310 | 0.320 | 1394.73 | 310 |
| 9 | 12/10/67 | 14:00 | 2580 | 27 | 1428.45 | 5850 | 6.125 | 1428.45 | 5850 |
| 10 | 12/10/67 | 14:00 | 2580 | 28 | 1438.47 | 2100 | 4.205 | 1438.47 | 2100 |
| 11 | 12/10/67 | 14:00 | 2580 | 29 | 1480.87 | 3450 | 3.645 | 1480.87 | 3450 |
| 12 | 12/10/67 | 14:00 | 2580 | 30 | 1505.16 | 140 | 0.140 | 1505.16 | 140 |
| 13 | 12/10/67 | 14:00 | 2580 | 31 | 1526.95 | 850 | 0.255 | 1526.95 | 850 |
| 14 | 12/10/67 | 14:00 | 2580 | 32 | 1561.44 | 2110 | 2.215 | 1561.44 | 2110 |
| 15 | 12/10/67 | 14:00 | 2580 | 33 | 1573.70 | 3470 | 3.630 | 1573.70 | 3470 |
| 16 | 12/10/67 | 14:00 | 2580 | 34 | 1593.09 | 1360 | 1.445 | 1593.09 | 1360 |
| 17 | 12/10/67 | 14:00 | 2580 | 35 | 1613.03 | 120 | 0.120 | 1613.03 | 120 |
| 18 | 12/10/67 | 14:00 | 2580 | 36 | 1635.57 | 940 | 1.035 | 1635.57 | 940 |
| 19 | 12/10/67 | 14:00 | 2580 | 37 | 1642.07 | 4210 | 4.400 | 1642.07 | 4210 |

| SUBJECT | DATE | TIME | SR | RC | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK AREA | NORMALIZED AREA |
|---------|----------|-------|------|----|----------------|-----------|-----------------|-----------|-----------------|
| 1 | 12/10/67 | 14:00 | 2580 | 17 | 1290.40 | 1080 | 0.820 | 1290.40 | 1080 |
| 2 | 12/10/67 | 14:00 | 2580 | 18 | 1310.82 | 680 | 0.517 | 1310.82 | 680 |
| 3 | 12/10/67 | 14:00 | 2580 | 19 | 1330.92 | 200 | 0.150 | 1330.92 | 200 |
| 4 | 12/10/67 | 14:00 | 2580 | 20 | 1363.70 | 4000 | 3.500 | 1363.70 | 4000 |
| 5 | 12/10/67 | 14:00 | 2580 | 21 | 1381.75 | 1400 | 1.000 | 1381.75 | 1400 |
| 6 | 12/10/67 | 14:00 | 2580 | 22 | 1417.44 | 4800 | 3.600 | 1417.44 | 4800 |
| 7 | 12/10/67 | 14:00 | 2580 | 23 | 1450.77 | 640 | 0.505 | 1450.77 | 640 |
| 8 | 12/10/67 | 14:00 | 2580 | 24 | 1470.45 | 680 | 0.520 | 1470.45 | 680 |
| 9 | 12/10/67 | 14:00 | 2580 | 25 | 1475.70 | 1400 | 1.094 | 1475.70 | 1400 |
| 10 | 12/10/67 | 14:00 | 2580 | 26 | 1500.31 | 720 | 0.555 | 1500.31 | 720 |
| 11 | 12/10/67 | 14:00 | 2580 | 27 | 1510.70 | 5000 | 4.400 | 1510.70 | 5000 |
| 12 | 12/10/67 | 14:00 | 2580 | 28 | 1530.14 | 1210 | 0.924 | 1530.14 | 1210 |
| 13 | 12/10/67 | 14:00 | 2580 | 29 | 1561.10 | 2000 | 1.500 | 1561.10 | 2000 |
| 14 | 12/10/67 | 14:00 | 2580 | 30 | 1590.70 | 700 | 0.501 | 1590.70 | 700 |
| 15 | 12/10/67 | 14:00 | 2580 | 31 | 1620.57 | 4500 | 3.930 | 1620.57 | 4500 |
| 16 | 12/10/67 | 14:00 | 2580 | 32 | 1643.03 | 1170 | 0.895 | 1643.03 | 1170 |

SUBJECT WHITE FEMALE EG
DATE 3-5-67
TOTAL INTENSIVE 50750

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.22 | 1420 | 3.243 | 21 | 1226.73 | 384 | 0.877 |
| 2 | 555.20 | 752 | 1.325 | 22 | 1239.93 | 2114 | 3.722 |
| 3 | 715.17 | 2674 | 4.624 | 23 | 1279.75 | 360 | 0.834 |
| 4 | 715.32 | 706 | 1.241 | 24 | 1291.09 | 504 | 0.888 |
| 5 | 805.03 | 1524 | 3.214 | 25 | 1311.03 | 1560 | 2.759 |
| 6 | 805.55 | 672 | 1.184 | 26 | 1338.99 | 1716 | 3.024 |
| 7 | 841.66 | 868 | 1.526 | 27 | 1353.27 | 260 | 0.987 |
| 8 | 914.09 | 576 | 1.015 | 28 | 1380.56 | 280 | 0.493 |
| 9 | 915.35 | 704 | 1.241 | 29 | 1392.57 | 676 | 1.191 |
| 10 | 964.32 | 640 | 1.128 | 30 | 1413.22 | 352 | 0.720 |
| 11 | 975.29 | 480 | 0.845 | 31 | 1427.77 | 880 | 1.521 |
| 12 | 975.54 | 768 | 1.353 | 32 | 1441.35 | 2640 | 4.652 |
| 13 | 1000.18 | 2752 | 4.849 | 33 | 1457.49 | 2520 | 4.441 |
| 14 | 1064.03 | 140 | 0.211 | 34 | 1482.76 | 2680 | 5.075 |
| 15 | 1092.82 | 228 | 0.421 | 35 | 1493.40 | 792 | 1.396 |
| 16 | 1105.44 | 492 | 0.867 | 36 | 1514.27 | 3600 | 6.344 |
| 17 | 1141.91 | 704 | 1.241 | 37 | 1536.09 | 4752 | 8.374 |
| 18 | 1154.70 | 72 | 0.127 | 38 | 1557.31 | 3328 | 5.864 |
| 19 | 1186.84 | 136 | 0.246 | 39 | 1566.57 | 2800 | 4.934 |
| 20 | 1210.96 | 288 | 0.507 | 40 | 1596.02 | 3344 | 5.893 |
| | | | | 41 | 1625.57 | 3332 | 5.871 |

SUBJECT WHITE FEMALE EG
DATE 3-5-67
TOTAL INTENSIVE 50750

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.22 | 1420 | 3.243 | 21 | 1226.73 | 384 | 0.877 |
| 2 | 555.20 | 752 | 1.325 | 22 | 1239.93 | 2114 | 3.722 |
| 3 | 715.17 | 2674 | 4.624 | 23 | 1279.75 | 360 | 0.834 |
| 4 | 715.32 | 706 | 1.241 | 24 | 1291.09 | 504 | 0.888 |
| 5 | 805.03 | 1524 | 3.214 | 25 | 1311.03 | 1560 | 2.759 |
| 6 | 805.55 | 672 | 1.184 | 26 | 1338.99 | 1716 | 3.024 |
| 7 | 841.66 | 868 | 1.526 | 27 | 1353.27 | 260 | 0.987 |
| 8 | 914.09 | 576 | 1.015 | 28 | 1380.56 | 280 | 0.493 |
| 9 | 915.35 | 704 | 1.241 | 29 | 1392.57 | 676 | 1.191 |
| 10 | 964.32 | 640 | 1.128 | 30 | 1413.22 | 352 | 0.720 |
| 11 | 975.29 | 480 | 0.845 | 31 | 1427.77 | 880 | 1.521 |
| 12 | 975.54 | 768 | 1.353 | 32 | 1441.35 | 2640 | 4.652 |
| 13 | 1000.18 | 2752 | 4.849 | 33 | 1457.49 | 2520 | 4.441 |
| 14 | 1064.03 | 140 | 0.211 | 34 | 1482.76 | 2680 | 5.075 |
| 15 | 1092.82 | 228 | 0.421 | 35 | 1493.40 | 792 | 1.396 |
| 16 | 1105.44 | 492 | 0.867 | 36 | 1514.27 | 3600 | 6.344 |
| 17 | 1141.91 | 704 | 1.241 | 37 | 1536.09 | 4752 | 8.374 |
| 18 | 1154.70 | 72 | 0.127 | 38 | 1557.31 | 3328 | 5.864 |
| 19 | 1186.84 | 136 | 0.246 | 39 | 1566.57 | 2800 | 4.934 |
| 20 | 1210.96 | 288 | 0.507 | 40 | 1596.02 | 3344 | 5.893 |
| | | | | 41 | 1625.57 | 3332 | 5.871 |

SUBJECT: Neuro Female PL

Hud

DATE: 9-20-66

TOTAL INTENSIVE: 9476

| PEAK | RETENTION TIME | MEAN AREA | MEAN INTENSIVE | PEAK | RETENTION TIME | MEAN AREA | MEAN INTENSIVE |
|------|----------------|-----------|----------------|------|----------------|-----------|----------------|
| 1 | 926.24 | 47.1 | 1.113 | 21 | 1236.32 | 300 | 0.8723 |
| 2 | 939.51 | 72 | 0.8723 | 22 | 1265.78 | 200 | 0.8723 |
| 3 | 721.71 | 54 | 1.3180 | 23 | 1274.01 | 384 | 0.8723 |
| 4 | 775.55 | 704 | 0.8723 | 24 | 1304.92 | 400 | 0.8723 |
| 5 | 807.12 | 1160 | 0.8723 | 25 | 1321.02 | 1440 | 0.8723 |
| 6 | 831.30 | 1398 | 0.8723 | 26 | 1345.67 | 87 | 0.8723 |
| 7 | 863.25 | 784 | 0.8723 | 27 | 1363.53 | 112 | 0.8723 |
| 8 | 914.95 | 312 | 0.8723 | 28 | 1378.34 | 720 | 0.8723 |
| 9 | 934.55 | 724 | 0.8723 | 29 | 1405.98 | 404 | 0.8723 |
| 10 | 973.54 | 1120 | 0.8723 | 30 | 1422.17 | 1482 | 0.8723 |
| 11 | 1012.34 | 544 | 0.8723 | 31 | 1452.24 | 300 | 0.8723 |
| 12 | 1023.49 | 575 | 0.8723 | 32 | 1464.51 | 680 | 0.8723 |
| 13 | 1035.80 | 124 | 0.8723 | 33 | 1474.25 | 330 | 0.8723 |
| 14 | 1061.87 | 1400 | 0.8723 | 34 | 1507.34 | 1152 | 0.8723 |
| 15 | 1127.21 | 504 | 0.8723 | 35 | 1523.52 | 1240 | 0.8723 |
| 16 | 1141.91 | 1256 | 0.8723 | 36 | 1534.71 | 300 | 0.8723 |
| 17 | 1147.57 | 1312 | 0.8723 | 37 | 1570.22 | 192 | 0.8723 |
| 18 | 1180.25 | 672 | 0.8723 | 38 | 1605.64 | 244 | 0.8723 |
| 19 | 1205.14 | 715 | 0.8723 | 39 | 1609.43 | 170 | 0.8723 |
| 20 | 1211.33 | 1051 | 0.8723 | 40 | 1613.54 | 400 | 0.8723 |
| | | | | 41 | 1625.57 | 760 | 0.8723 |

SUBJECT: Neuro Female PL

PL

DATE: 9-20-66

TOTAL INTENSIVE: 9476

| PEAK | RETENTION TIME | MEAN AREA | MEAN INTENSIVE | PEAK | RETENTION TIME | MEAN AREA | MEAN INTENSIVE |
|------|----------------|-----------|----------------|------|----------------|-----------|----------------|
| 1 | 926.24 | 108 | 0.178 | 21 | 1243.00 | 5376 | 5.718 |
| 2 | 939.51 | 144 | 0.178 | 22 | 1274.01 | 2880 | 3.078 |
| 3 | 721.71 | 212 | 0.178 | 23 | 1310.35 | 1280 | 1.359 |
| 4 | 775.55 | 445 | 0.178 | 24 | 1324.21 | 3328 | 3.534 |
| 5 | 807.12 | 4288 | 0.178 | 25 | 1348.20 | 280 | 0.297 |
| 6 | 831.30 | 2645 | 0.178 | 26 | 1370.23 | 800 | 0.844 |
| 7 | 863.25 | 1200 | 0.178 | 27 | 1374.01 | 704 | 0.748 |
| 8 | 914.95 | 640 | 0.178 | 28 | 1395.37 | 96 | 0.102 |
| 9 | 934.55 | 540 | 0.178 | 29 | 1410.25 | 760 | 0.807 |
| 10 | 973.54 | 2615 | 0.178 | 30 | 1424.77 | 1924 | 2.013 |
| 11 | 1012.34 | 1376 | 0.178 | 31 | 1440.34 | 84 | 0.089 |
| 12 | 1023.49 | 925 | 0.178 | 32 | 1465.31 | 1020 | 1.083 |
| 13 | 1035.80 | 5072 | 0.178 | 33 | 1485.85 | 280 | 0.276 |
| 14 | 1061.87 | 5942 | 0.178 | 34 | 1510.47 | 1800 | 1.911 |
| 15 | 1127.21 | 11262 | 0.178 | 35 | 1525.70 | 2024 | 2.149 |
| 16 | 1141.91 | 5720 | 0.178 | 36 | 1540.43 | 432 | 0.454 |
| 17 | 1147.57 | 4415 | 0.178 | 37 | 1556.01 | 200 | 0.214 |
| 18 | 1180.25 | 3940 | 0.178 | 38 | 1587.11 | 252 | 0.268 |
| 19 | 1205.14 | 5528 | 0.178 | 39 | 1607.05 | 480 | 0.510 |
| 20 | 1211.33 | | | 40 | 1625.57 | 972 | 1.034 |

SUBJECT RESUBMIT DATE
DATE 27-05-67
TOTAL INTENSIVE 1705.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 5.05.03 | 2100 | 0.076 | 24 | 16.00.18 | 2800 | 0.071 |
| 2 | 5.13.72 | 1800 | 0.067 | 25 | 16.07.18 | 3600 | 0.074 |
| 3 | 5.23.55 | 1600 | 0.052 | 26 | 17.02.07 | 4000 | 0.051 |
| 4 | 6.01.51 | 700 | 0.027 | 27 | 17.17.08 | 4400 | 0.051 |
| 5 | 7.01.01 | 700 | 0.027 | 28 | 17.55.02 | 4200 | 0.051 |
| 6 | 7.08.15 | 2700 | 0.097 | 29 | 18.03.42 | 5100 | 0.053 |
| 7 | 8.51.40 | 3100 | 0.105 | 30 | 18.08.38 | 1600 | 0.045 |
| 8 | 8.58.08 | 500 | 0.018 | 31 | 18.08.76 | 2600 | 0.060 |
| 9 | 9.47.23 | 1100 | 0.037 | 32 | 18.21.22 | 2800 | 0.071 |
| 10 | 9.53.37 | 200 | 0.007 | 33 | 18.30.25 | 3600 | 0.084 |
| 11 | 9.58.55 | 1200 | 0.042 | 34 | 18.50.06 | 800 | 0.021 |
| 12 | 9.58.18 | 500 | 0.018 | 35 | 18.53.16 | 2500 | 0.064 |
| 13 | 9.58.53 | 500 | 0.018 | 36 | 18.58.36 | 2500 | 0.064 |
| 14 | 10.12.38 | 700 | 0.027 | 37 | 19.09.09 | 2400 | 0.062 |
| 15 | 10.20.49 | 1500 | 0.052 | 38 | 19.23.30 | 3200 | 0.080 |
| 16 | 10.53.48 | 1000 | 0.037 | 39 | 19.37.05 | 2900 | 0.075 |
| 17 | 10.58.90 | 4000 | 0.135 | 40 | 19.41.08 | 3000 | 0.082 |
| 18 | 10.58.33 | 700 | 0.027 | 41 | 19.53.77 | 2100 | 0.068 |
| 19 | 11.41.91 | 1200 | 0.042 | 42 | 19.56.28 | 1400 | 0.045 |
| 20 | 11.47.00 | 4500 | 0.155 | 43 | 19.59.18 | 2100 | 0.075 |
| 21 | 11.47.45 | 500 | 0.018 | 44 | 19.58.38 | 400 | 0.012 |
| 22 | 12.00.26 | 1000 | 0.037 | 45 | 19.58.32 | 3000 | 0.082 |
| 23 | 12.35.90 | 1300 | 0.045 | 46 | 19.58.57 | 2600 | 0.071 |

SUBJECT 44116 PALE HN
DATE 25-05-66
TOTAL INTENSIVE 2105.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 4.05.55 | 400 | 0.018 | 19 | 11.42.41 | 3200 | 0.074 |
| 2 | 5.05.32 | 2700 | 0.125 | 20 | 12.00.04 | 1000 | 0.046 |
| 3 | 5.31.12 | 1700 | 0.076 | 21 | 12.37.25 | 2900 | 0.067 |
| 4 | 5.51.69 | 300 | 0.013 | 22 | 12.53.58 | 500 | 0.022 |
| 5 | 6.22.00 | 1800 | 0.082 | 23 | 12.58.72 | 800 | 0.036 |
| 6 | 7.52.32 | 5500 | 0.250 | 24 | 12.58.16 | 700 | 0.031 |
| 7 | 8.42.23 | 2100 | 0.095 | 25 | 13.22.19 | 3700 | 0.170 |
| 8 | 8.58.63 | 2112 | 0.096 | 26 | 13.46.34 | 3200 | 0.146 |
| 9 | 9.17.62 | 200 | 0.009 | 27 | 13.46.26 | 1100 | 0.051 |
| 10 | 9.57.51 | 1000 | 0.045 | 28 | 13.46.12 | 600 | 0.027 |
| 11 | 9.58.36 | 1400 | 0.062 | 29 | 13.58.10 | 1400 | 0.062 |
| 12 | 9.58.19 | 1215 | 0.054 | 30 | 14.12.70 | 300 | 0.013 |
| 13 | 10.03.02 | 400 | 0.018 | 31 | 14.31.35 | 1900 | 0.087 |
| 14 | 10.06.76 | 1100 | 0.049 | 32 | 14.54.59 | 600 | 0.027 |
| 15 | 10.07.41 | 600 | 0.027 | 33 | 14.50.83 | 800 | 0.036 |
| 16 | 11.01.45 | 900 | 0.041 | 34 | 14.51.51 | 2100 | 0.098 |
| 17 | 11.47.01 | 300 | 0.013 | 35 | 15.27.81 | 1400 | 0.064 |
| 18 | 11.58.30 | 800 | 0.036 | 36 | 16.03.50 | 2400 | 0.102 |
| | | | | 37 | 16.25.57 | 2400 | 0.108 |

SUBJECT WHITE MALE HN
DATE 7-9-50
TOTAL INTENSITY 15976

5724

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 527.85 | 65 | 0.445 | 19 | 1152.33 | 40 | 0.248 |
| 2 | 569.62 | 110 | 0.569 | 20 | 1169.37 | 200 | 1.035 |
| 3 | 610.02 | 72 | 0.373 | 21 | 1199.88 | 90 | 0.466 |
| 4 | 652.85 | 270 | 1.397 | 22 | 1219.97 | 120 | 0.621 |
| 5 | 666.65 | 312 | 1.616 | 23 | 1236.43 | 490 | 2.535 |
| 6 | 745.20 | 563 | 2.894 | 24 | 1264.50 | 104 | 0.538 |
| 7 | 803.91 | 1763 | 9.107 | 25 | 1279.78 | 64 | 0.331 |
| 8 | 841.50 | 243 | 11.591 | 26 | 1307.40 | 496 | 2.566 |
| 9 | 872.87 | 483 | 2.484 | 27 | 1334.02 | 120 | 0.621 |
| 10 | 895.04 | 704 | 3.643 | 28 | 1351.04 | 486 | 2.515 |
| 11 | 927.56 | 1973 | 9.935 | 29 | 1372.52 | 150 | 0.776 |
| 12 | 942.98 | 672 | 3.477 | 30 | 1387.33 | 42 | 0.217 |
| 13 | 984.75 | 415 | 2.153 | 31 | 1416.30 | 930 | 4.812 |
| 14 | 993.30 | 95 | 0.497 | 32 | 1470.42 | 1140 | 5.859 |
| 15 | 1004.91 | 848 | 4.385 | 33 | 1486.06 | 91 | 0.471 |
| 16 | 1139.19 | 440 | 2.484 | 34 | 1523.18 | 2700 | 13.971 |
| 17 | 1081.58 | 183 | 0.931 | 35 | 1555.73 | 180 | 0.931 |
| 18 | 1127.91 | 490 | 2.073 | 36 | 1595.52 | 144 | 0.745 |
| | | | | 37 | 1625.57 | 125 | 0.647 |

SUBJECT WHITE MALE HN
DATE 2-16-56
TOTAL INTENSITY 23270

846

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 569.47 | 168 | 0.722 | 19 | 1203.05 | 210 | 0.928 |
| 2 | 610.02 | 220 | 0.945 | 20 | 1222.97 | 504 | 2.166 |
| 3 | 652.44 | 420 | 1.805 | 21 | 1235.10 | 1440 | 6.186 |
| 4 | 725.70 | 280 | 1.203 | 22 | 1257.41 | 160 | 0.668 |
| 5 | 745.01 | 220 | 0.945 | 23 | 1276.39 | 320 | 1.375 |
| 6 | 845.17 | 803 | 3.438 | 24 | 1292.16 | 960 | 4.125 |
| 7 | 868.66 | 1735 | 7.460 | 25 | 1311.32 | 1100 | 4.727 |
| 8 | 894.69 | 688 | 2.957 | 26 | 1343.39 | 476 | 2.046 |
| 9 | 917.79 | 732 | 3.144 | 27 | 1365.82 | 80 | 0.344 |
| 10 | 936.55 | 302 | 1.295 | 28 | 1405.60 | 1350 | 5.801 |
| 11 | 977.95 | 240 | 1.031 | 29 | 1432.32 | 456 | 1.900 |
| 12 | 1001.05 | 345 | 1.450 | 30 | 1459.63 | 1560 | 6.704 |
| 13 | 1015.12 | 912 | 3.919 | 31 | 1473.37 | 280 | 1.203 |
| 14 | 1050.05 | 123 | 0.516 | 32 | 1507.60 | 1920 | 8.251 |
| 15 | 1072.85 | 672 | 2.888 | 33 | 1534.69 | 160 | 0.688 |
| 16 | 1097.93 | 303 | 1.289 | 34 | 1578.95 | 180 | 0.774 |
| 17 | 1121.91 | 640 | 2.750 | 35 | 1601.37 | 150 | 0.645 |
| 18 | 1157.03 | 352 | 1.513 | 36 | 1625.57 | 250 | 1.074 |

SUBJECT: 4411111111 AD
DATE: 10-10-66
TOTAL INTENSITY: 12

661

| PEAK | RETENTION TIME | AREA | AREA PERCENT | PEAK AREA | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|------|--------------|-----------|----------------|-----------|-----------------|
| 1 | 9.75.07 | 13.1 | 1.00 | 100 | 12.60.77 | 100 | 6.274 |
| 2 | 9.75.28 | 13.1 | 1.00 | 100 | 12.77.56 | 100 | 1.051 |
| 3 | 10.22.58 | 14.0 | 1.00 | 100 | 13.06.22 | 100 | 0.450 |
| 4 | 10.31.56 | 14.0 | 1.00 | 100 | 13.25.65 | 100 | 7.009 |
| 5 | 10.31.51 | 14.0 | 1.00 | 100 | 13.47.61 | 100 | 0.876 |
| 6 | 10.31.57 | 14.0 | 1.00 | 100 | 13.64.91 | 100 | 1.183 |
| 7 | 10.31.52 | 14.0 | 1.00 | 100 | 13.82.50 | 100 | 16.295 |
| 8 | 10.31.55 | 14.0 | 1.00 | 100 | 14.06.76 | 100 | 2.103 |
| 9 | 10.31.52 | 14.0 | 1.00 | 100 | 14.25.27 | 100 | 16.121 |
| 10 | 10.31.19 | 14.0 | 1.00 | 100 | 14.47.76 | 100 | 0.789 |
| 11 | 10.31.75 | 14.0 | 1.00 | 100 | 14.64.13 | 100 | 2.141 |
| 12 | 10.31.26 | 14.0 | 1.00 | 100 | 14.72.61 | 100 | 1.843 |
| 13 | 10.31.23 | 14.0 | 1.00 | 100 | 15.07.26 | 100 | 1.917 |
| 14 | 11.11.51 | 14.0 | 1.00 | 100 | 15.25.55 | 100 | 4.408 |
| 15 | 11.17.74 | 14.0 | 1.00 | 100 | 15.35.23 | 100 | 1.971 |
| 16 | 11.17.51 | 14.0 | 1.00 | 100 | 15.52.48 | 100 | 0.657 |
| 17 | 12.04.41 | 14.0 | 1.00 | 100 | 15.78.69 | 100 | 2.793 |
| 18 | 12.04.75 | 14.0 | 1.00 | 100 | 16.01.20 | 100 | 1.862 |
| 19 | 12.35.97 | 14.0 | 1.00 | 100 | 16.25.57 | 100 | 3.614 |

40

976

40

976

| PEAK | RETENTION TIME | AREA | AREA PERCENT | PEAK AREA | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|------|--------------|-----------|----------------|-----------|-----------------|
| 1 | 9.75.70 | 18.5 | 1.00 | 100 | 12.33.99 | 100 | 2.418 |
| 2 | 9.75.07 | 18.5 | 1.00 | 100 | 12.60.77 | 100 | 0.757 |
| 3 | 10.22.58 | 18.5 | 1.00 | 100 | 12.77.56 | 100 | 0.368 |
| 4 | 10.31.56 | 18.5 | 1.00 | 100 | 13.05.84 | 100 | 1.151 |
| 5 | 10.31.51 | 18.5 | 1.00 | 100 | 13.17.60 | 100 | 0.757 |
| 6 | 10.31.57 | 18.5 | 1.00 | 100 | 13.26.00 | 100 | 1.798 |
| 7 | 10.31.52 | 18.5 | 1.00 | 100 | 13.45.17 | 100 | 0.368 |
| 8 | 10.31.55 | 18.5 | 1.00 | 100 | 13.69.44 | 100 | 0.258 |
| 9 | 10.31.52 | 18.5 | 1.00 | 100 | 13.85.75 | 100 | 1.766 |
| 10 | 10.31.19 | 18.5 | 1.00 | 100 | 14.07.07 | 100 | 1.703 |
| 11 | 10.31.75 | 18.5 | 1.00 | 100 | 14.26.93 | 100 | 3.306 |
| 12 | 10.31.26 | 18.5 | 1.00 | 100 | 14.51.53 | 100 | 0.520 |
| 13 | 10.31.23 | 18.5 | 1.00 | 100 | 14.66.73 | 100 | 4.111 |
| 14 | 10.31.59 | 18.5 | 1.00 | 100 | 15.07.63 | 100 | 3.785 |
| 15 | 11.11.91 | 18.5 | 1.00 | 100 | 15.24.50 | 100 | 3.406 |
| 16 | 11.17.97 | 18.5 | 1.00 | 100 | 15.31.61 | 100 | 3.259 |
| 17 | 11.17.25 | 18.5 | 1.00 | 100 | 15.71.57 | 100 | 2.460 |
| 18 | 12.03.02 | 18.5 | 1.00 | 100 | 15.80.57 | 100 | 1.430 |
| 19 | 12.24.16 | 18.5 | 1.00 | 100 | 15.99.94 | 100 | 1.892 |
| | | | | | 16.25.57 | 100 | 7.738 |

SUBJECT WHITL GALL AD 41
 DATE 12-13-66 8541-
 TOTAL INTENSITY= MEAN INTENSITY= 2375.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.85 | 156. | 0.153 | 19 | 1155.24 | 100. | 0.117 |
| 2 | 605.00 | 275. | 0.325 | 20 | 1176.13 | 240. | 0.281 |
| 3 | 698.39 | 132. | 0.155 | 21 | 1205.76 | 70. | 0.082 |
| 4 | 717.15 | 1102. | 1.344 | 22 | 1273.95 | 526. | 0.618 |
| 5 | 775.86 | 304. | 0.355 | 23 | 1292.20 | 900. | 1.024 |
| 6 | 791.81 | 382. | 0.412 | 24 | 1280.41 | 288. | 0.337 |
| 7 | 815.53 | 1455. | 1.705 | 25 | 1296.65 | 208. | 0.244 |
| 8 | 882.20 | 175. | 0.206 | 26 | 1316.83 | 1560. | 1.826 |
| 9 | 905.34 | 175. | 0.206 | 27 | 1343.42 | 216. | 0.253 |
| 10 | 958.86 | 117. | 0.131 | 28 | 1377.84 | 182. | 0.213 |
| 11 | 973.32 | 288. | 0.337 | 29 | 1406.06 | 840. | 0.983 |
| 12 | 984.54 | 275. | 0.323 | 30 | 1423.15 | 8112. | 9.497 |
| 13 | 1002.81 | 400. | 0.466 | 31 | 1461.87 | 5336. | 6.247 |
| 14 | 1028.56 | 576. | 0.674 | 32 | 1505.48 | 3108. | 3.639 |
| 15 | 1055.25 | 448. | 0.524 | 33 | 1525.70 | 13552. | 15.866 |
| 16 | 1074.84 | 280. | 0.326 | 34 | 1567.92 | 22400. | 26.225 |
| 17 | 1097.15 | 168. | 0.197 | 35 | 1602.23 | 8832. | 10.340 |
| 18 | 1121.91 | 656. | 0.764 | 36 | 1625.57 | 9856. | 11.539 |

SUBJECT WHITL GALL AD 42
 DATE 12-12-66 64036.
 TOTAL INTENSITY= MEAN INTENSITY= 1217.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 408.19 | 44. | 0.064 | 20 | 1205.86 | 72. | 0.104 |
| 2 | 525.34 | 112. | 0.162 | 21 | 1227.45 | 800. | 1.246 |
| 3 | 602.00 | 72. | 0.104 | 22 | 1237.83 | 2248. | 3.662 |
| 4 | 657.92 | 76. | 0.110 | 23 | 1266.83 | 240. | 0.348 |
| 5 | 734.36 | 1106. | 1.599 | 24 | 1295.69 | 500. | 0.724 |
| 6 | 791.81 | 515. | 0.747 | 25 | 1309.79 | 804. | 1.165 |
| 7 | 815.53 | 1475. | 2.136 | 26 | 1320.03 | 1480. | 2.115 |
| 8 | 895.20 | 272. | 0.394 | 27 | 1345.28 | 400. | 0.574 |
| 9 | 912.51 | 552. | 0.861 | 28 | 1362.36 | 720. | 1.043 |
| 10 | 932.68 | 72. | 0.104 | 29 | 1379.56 | 5016. | 7.266 |
| 11 | 967.98 | 128. | 0.185 | 30 | 1406.32 | 3296. | 4.774 |
| 12 | 984.54 | 128. | 0.185 | 31 | 1427.64 | 1244. | 1.785 |
| 13 | 996.17 | 200. | 0.290 | 32 | 1463.19 | 11840. | 17.150 |
| 14 | 1007.34 | 276. | 0.400 | 33 | 1507.82 | 3600. | 5.226 |
| 15 | 1025.24 | 456. | 0.661 | 34 | 1528.65 | 10676. | 15.459 |
| 16 | 1058.79 | 312. | 0.452 | 35 | 1554.84 | 4592. | 7.231 |
| 17 | 1100.05 | 240. | 0.348 | 36 | 1588.27 | 9200. | 13.326 |
| 18 | 1121.91 | 1104. | 1.599 | 37 | 1601.90 | 2304. | 3.337 |
| 19 | 1178.19 | 288. | 0.417 | 38 | 1625.57 | 1960. | 2.839 |

SUBJECT DATE 12-19-56 AD 12-19-56 EXP-2 42

TOTAL INTENSITY 1-16.7. MEAN INTENSITY 478.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.25 | 542. | 3.534 | 20 | 1217.16 | 378. | 2.026 |
| 2 | 500.60 | 164. | 0.903 | 21 | 1248.00 | 142. | 1.057 |
| 3 | 717.15 | 608. | 3.347 | 22 | 1266.29 | 143. | 0.767 |
| 4 | 765.05 | 512. | 2.816 | 23 | 1274.76 | 96. | 0.528 |
| 5 | 804.01 | 440. | 1.921 | 24 | 1294.15 | 288. | 1.585 |
| 6 | 835.84 | 72. | 0.395 | 25 | 1318.70 | 94. | 0.886 |
| 7 | 890.22 | 112. | 0.617 | 26 | 1338.88 | 144. | 0.793 |
| 8 | 905.34 | 395. | 2.180 | 27 | 1360.40 | 72. | 0.346 |
| 9 | 945.17 | 166. | 0.925 | 28 | 1376.79 | 646. | 3.556 |
| 10 | 952.55 | 152. | 0.837 | 29 | 1401.53 | 396. | 2.180 |
| 11 | 965.12 | 256. | 1.407 | 30 | 1416.38 | 183. | 8.994 |
| 12 | 1011.74 | 400. | 2.202 | 31 | 1454.90 | 506. | 2.765 |
| 13 | 1040.46 | 348. | 1.915 | 32 | 1465.50 | 115. | 6.341 |
| 14 | 1065.67 | 89. | 0.484 | 33 | 1502.32 | 1480. | 8.147 |
| 15 | 1085.12 | 464. | 2.554 | 34 | 1525.27 | 2436. | 13.409 |
| 16 | 1121.57 | 108. | 0.594 | 35 | 1578.31 | 876. | 4.932 |
| 17 | 1155.70 | 175. | 0.969 | 36 | 1587.55 | 300. | 1.982 |
| 18 | 1182.69 | 50. | 0.275 | 37 | 1599.01 | 424. | 1.387 |
| 19 | 1204.35 | 580. | 3.193 | 38 | 1625.57 | 646. | 3.556 |

SUBJECT DATE 12-19-56 AD 12-19-56 EXP-2 44

TOTAL INTENSITY 84078. MEAN INTENSITY 2289.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.22 | 480. | 0.567 | 19 | 1179.80 | 3480. | 4.110 |
| 2 | 613.26 | 260. | 0.307 | 20 | 1223.95 | 3072. | 3.628 |
| 3 | 678.11 | 260. | 0.307 | 21 | 1241.72 | 1264. | 1.512 |
| 4 | 723.65 | 1160. | 1.370 | 22 | 1272.42 | 1232. | 1.455 |
| 5 | 768.52 | 160. | 0.189 | 23 | 1282.65 | 288. | 0.340 |
| 6 | 808.97 | 325. | 0.385 | 24 | 1324.79 | 6016. | 7.105 |
| 7 | 827.80 | 2412. | 2.848 | 25 | 1356.47 | 364. | 0.433 |
| 8 | 865.55 | 129. | 0.151 | 26 | 1388.30 | 4864. | 5.744 |
| 9 | 898.27 | 580. | 0.687 | 27 | 1427.53 | 8400. | 9.920 |
| 10 | 918.75 | 960. | 1.134 | 28 | 1470.42 | 5440. | 6.424 |
| 11 | 960.58 | 528. | 0.624 | 29 | 1483.71 | 1440. | 1.701 |
| 12 | 978.71 | 304. | 0.359 | 30 | 1495.77 | 576. | 0.680 |
| 13 | 995.41 | 2160. | 2.551 | 31 | 1512.90 | 528. | 0.624 |
| 14 | 1035.17 | 1824. | 2.154 | 32 | 1528.48 | 9360. | 11.054 |
| 15 | 1089.15 | 2968. | 3.488 | 33 | 1548.25 | 1152. | 1.360 |
| 16 | 1093.69 | 1920. | 2.267 | 34 | 1570.52 | 1344. | 1.587 |
| 17 | 1121.91 | 9600. | 11.337 | 35 | 1581.20 | 960. | 1.143 |
| 18 | 1144.10 | 544. | 0.642 | 36 | 1604.69 | 1800. | 0.213 |
| | | | | 37 | 1625.57 | 7200. | 8.503 |

SUBJECT INDIAN MALE RS 45
DATE 11-11-58
TOTAL INTENSITY 50000
VEGETATION 13520

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 474.04 | 478 | 0.480 | 23 | 1177.08 | 1080 | 1.015 |
| 2 | 512.23 | 180 | 0.469 | 24 | 1206.79 | 480 | 0.484 |
| 3 | 622.01 | 80 | 0.101 | 25 | 1221.52 | 1100 | 1.055 |
| 4 | 641.89 | 440 | 0.719 | 26 | 1235.17 | 163 | 2.745 |
| 5 | 745.03 | 1320 | 2.216 | 27 | 1268.40 | 1088 | 1.828 |
| 6 | 765.32 | 246 | 0.417 | 28 | 1289.50 | 280 | 0.471 |
| 7 | 767.89 | 192 | 0.323 | 29 | 1305.85 | 280 | 0.471 |
| 8 | 833.82 | 732 | 1.230 | 30 | 1320.03 | 1232 | 2.070 |
| 9 | 865.68 | 440 | 0.417 | 31 | 1349.75 | 440 | 0.444 |
| 10 | 893.28 | 746 | 0.417 | 32 | 1362.61 | 264 | 0.444 |
| 11 | 917.69 | 672 | 1.129 | 33 | 1377.45 | 1364 | 2.292 |
| 12 | 935.36 | 300 | 0.548 | 34 | 1408.75 | 588 | 0.986 |
| 13 | 964.94 | 320 | 0.536 | 35 | 1421.10 | 2112 | 3.544 |
| 14 | 982.57 | 300 | 0.524 | 36 | 1444.65 | 416 | 0.634 |
| 15 | 995.02 | 420 | 0.370 | 37 | 1463.04 | 3096 | 5.203 |
| 16 | 1001.94 | 536 | 0.901 | 38 | 1476.43 | 1464 | 2.360 |
| 17 | 1006.52 | 536 | 0.901 | 39 | 1489.01 | 952 | 1.600 |
| 18 | 1034.71 | 640 | 1.076 | 40 | 1509.10 | 640 | 1.076 |
| 19 | 1061.88 | 560 | 0.941 | 41 | 1522.09 | 3240 | 5.443 |
| 20 | 1094.34 | 720 | 1.210 | 42 | 1542.11 | 1392 | 2.3420 |
| 21 | 1111.91 | 800 | 1.344 | 43 | 1571.80 | 7424 | 12.472 |
| 22 | 1145.33 | 360 | 0.600 | 44 | 1625.57 | 6536 | 10.984 |

SUBJECT INDIAN MALE RS 46
DATE 12-5-58
TOTAL INTENSITY 67240
NEAR INTENSITY 1401

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 407.23 | 104 | 0.155 | 27 | 1206.00 | 940 | 1.398 |
| 2 | 474.22 | 324 | 0.482 | 28 | 1227.13 | 1076 | 1.570 |
| 3 | 525.42 | 140 | 0.208 | 29 | 1240.38 | 3864 | 5.747 |
| 4 | 655.20 | 244 | 0.363 | 30 | 1265.79 | 1084 | 1.582 |
| 5 | 717.74 | 944 | 1.404 | 31 | 1286.98 | 1360 | 2.023 |
| 6 | 735.04 | 940 | 1.428 | 32 | 1310.74 | 1776 | 2.641 |
| 7 | 805.03 | 1824 | 2.713 | 33 | 1337.18 | 4160 | 6.105 |
| 8 | 815.62 | 480 | 0.713 | 34 | 1351.70 | 672 | 0.999 |
| 9 | 857.48 | 1344 | 1.997 | 35 | 1391.27 | 200 | 0.291 |
| 10 | 883.62 | 4160 | 6.212 | 36 | 1400.55 | 160 | 0.238 |
| 11 | 905.87 | 480 | 0.713 | 37 | 1416.34 | 720 | 1.071 |
| 12 | 927.81 | 1848 | 2.746 | 38 | 1439.91 | 1536 | 2.284 |
| 13 | 964.32 | 3760 | 5.592 | 39 | 1454.84 | 440 | 0.654 |
| 14 | 995.41 | 1256 | 1.868 | 40 | 1467.35 | 672 | 0.999 |
| 15 | 1003.18 | 1144 | 1.701 | 41 | 1479.58 | 392 | 0.583 |
| 16 | 1034.64 | 2500 | 3.714 | 42 | 1515.30 | 1200 | 1.874 |
| 17 | 1071.41 | 4416 | 6.568 | 43 | 1541.91 | 5880 | 8.745 |
| 18 | 1102.56 | 1404 | 2.084 | 44 | 1568.55 | 240 | 0.357 |
| 19 | 1111.91 | 3476 | 5.163 | 45 | 1586.20 | 560 | 0.833 |
| 20 | 1122.64 | 1360 | 2.023 | 46 | 1596.35 | 600 | 0.892 |
| 21 | 1145.03 | 1764 | 2.623 | 47 | 1625.57 | 648 | 0.954 |

SUBJECT INDIAN, PAK

173.

TOTAL INTENSITY

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 467.88 | 68. | 0.981 | 21 | 1113.91 | 7108. | 5.879 |
| 2 | 474.02 | 56. | 0.791 | 22 | 1112.61 | 70. | 0.989 |
| 3 | 503.85 | 92. | 0.735 | 23 | 1205.80 | 50. | 0.707 |
| 4 | 510.44 | 64. | 0.961 | 24 | 1226.98 | 130. | 1.857 |
| 5 | 525.17 | 58. | 0.791 | 25 | 1239.80 | 160. | 1.979 |
| 6 | 725.84 | 36. | 0.427 | 26 | 1265.44 | 76. | 1.018 |
| 7 | 759.79 | 140. | 1.979 | 27 | 1274.02 | 50. | 0.791 |
| 8 | 803.34 | 132. | 1.865 | 28 | 1308.90 | 70. | 0.989 |
| 9 | 822.91 | 372. | 5.242 | 29 | 1330.45 | 120. | 1.696 |
| 10 | 855.55 | 236. | 3.279 | 30 | 1347.85 | 104. | 1.470 |
| 11 | 885.75 | 198. | 2.795 | 31 | 1370.75 | 30. | 0.424 |
| 12 | 916.25 | 394. | 4.296 | 32 | 1381.43 | 90. | 1.272 |
| 13 | 931.29 | 180. | 2.221 | 33 | 1408.72 | 100. | 1.413 |
| 14 | 975.70 | 176. | 2.487 | 34 | 1428.31 | 170. | 2.402 |
| 15 | 1152.31 | 682. | 9.904 | 35 | 1458.47 | 45. | 0.639 |
| 16 | 1166.01 | 168. | 2.374 | 36 | 1465.89 | 144. | 2.035 |
| 17 | 1002.34 | 64. | 0.904 | 37 | 1480.39 | 180. | 2.544 |
| 18 | 1015.09 | 572. | 8.142 | 38 | 1507.82 | 140. | 1.979 |
| 19 | 1063.11 | 320. | 4.522 | 39 | 1520.24 | 540. | 7.631 |
| 20 | 1102.35 | 84. | 1.187 | 40 | 1578.89 | 130. | 1.922 |
| | | | | 41 | 1625.57 | 600. | 8.679 |

SUBJECT WHITE, PAK

2185.

TOTAL INTENSITY

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 445.78 | 80. | 8.085 | 22 | 1273.50 | 7108. | 7.648 |
| 2 | 480.40 | 84. | 7.089 | 23 | 1310.48 | 1232. | 1.311 |
| 3 | 528.58 | 144. | 6.152 | 24 | 1325.46 | 4100. | 4.427 |
| 4 | 593.54 | 52. | 8.035 | 25 | 1341.15 | 1400. | 1.532 |
| 5 | 725.35 | 1152. | 1.226 | 26 | 1375.23 | 4800. | 5.108 |
| 6 | 805.48 | 624. | 8.664 | 27 | 1381.58 | 4032. | 4.291 |
| 7 | 826.24 | 1342. | 1.481 | 28 | 1409.54 | 2040. | 2.171 |
| 8 | 891.25 | 1470. | 2.043 | 29 | 1425.96 | 800. | 0.851 |
| 9 | 905.15 | 722. | 0.765 | 30 | 1440.14 | 1140. | 1.217 |
| 10 | 929.67 | 720. | 0.765 | 31 | 1453.94 | 704. | 0.749 |
| 11 | 951.59 | 1536. | 1.635 | 32 | 1468.75 | 2880. | 3.065 |
| 12 | 980.35 | 1800. | 1.915 | 33 | 1485.61 | 11400. | 12.132 |
| 13 | 1001.55 | 1008. | 1.073 | 34 | 1510.53 | 1008. | 1.075 |
| 14 | 1041.67 | 7040. | 7.492 | 35 | 1524.05 | 1848. | 1.957 |
| 15 | 1122.48 | 4800. | 5.118 | 36 | 1541.55 | 616. | 0.656 |
| 16 | 1111.91 | 2912. | 3.099 | 37 | 1555.82 | 936. | 0.996 |
| 17 | 1144.20 | 1280. | 1.362 | 38 | 1574.04 | 3224. | 3.431 |
| 18 | 1171.95 | 4360. | 4.511 | 39 | 1586.08 | 3680. | 3.878 |
| 19 | 1207.79 | 4604. | 4.904 | 40 | 1598.74 | 280. | 0.298 |
| 20 | 1250.14 | 1440. | 1.532 | 41 | 1602.72 | 320. | 0.341 |
| 21 | 1251.54 | 1600. | 1.793 | 42 | 1611.40 | 404. | 0.919 |
| | | | | 43 | 1625.57 | 2720. | 2.895 |

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SUBJECT WHITE FEMALE DC

DATE 7-15-57

TOTAL INTENSIVE 375000

MEAN INTENSIVE 9000

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PLAN | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 135.55 | 212. | 0.054 | 24 | 142.45 | 1292. | 3.436 |
| 2 | 414.34 | 170. | 0.042 | 23 | 1267.49 | 14592. | 3.859 |
| 3 | 527.75 | 435. | 0.112 | 24 | 1278.40 | 16704. | 4.418 |
| 4 | 675.27 | 420. | 0.111 | 25 | 1296.53 | 3640. | 1.016 |
| 5 | 730.82 | 1440. | 0.381 | 26 | 1331.55 | 8488. | 2.234 |
| 6 | 747.42 | 2730. | 0.719 | 27 | 1350.95 | 1568. | 0.415 |
| 7 | 847.15 | 1655. | 0.438 | 28 | 1383.01 | 5528. | 1.472 |
| 8 | 847.90 | 1468. | 0.387 | 29 | 1392.26 | 15240. | 4.031 |
| 9 | 851.22 | 4815. | 0.745 | 30 | 1412.04 | 2648. | 0.695 |
| 10 | 945.34 | 1555. | 0.760 | 31 | 1439.53 | 1450. | 0.381 |
| 11 | 945.33 | 2684. | 0.711 | 32 | 1449.35 | 1280. | 0.339 |
| 12 | 951.04 | 6976. | 0.787 | 33 | 1484.23 | 3040. | 0.804 |
| 13 | 965.31 | 6950. | 2.570 | 34 | 1496.14 | 11520. | 3.047 |
| 14 | 995.19 | 35840. | 7.475 | 35 | 1524.62 | 1232. | 0.326 |
| 15 | 1025.24 | 26840. | 7.103 | 36 | 1534.89 | 784. | 0.207 |
| 16 | 1076.43 | 5375. | 8.548 | 37 | 1550.10 | 1716. | 0.454 |
| 17 | 1141.71 | 14520. | 3.047 | 38 | 1568.63 | 924. | 0.244 |
| 18 | 1151.61 | 25536. | 6.754 | 39 | 1586.54 | 3864. | 1.022 |
| 19 | 1171.55 | 30648. | 8.137 | 40 | 1598.58 | 2484. | 0.784 |
| 20 | 1184.74 | 25525. | 6.754 | 41 | 1625.57 | 2160. | 0.571 |
| 21 | 1215.70 | 45704. | 10.765 | 42 | 1636.33 | 1976. | 0.523 |

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SUBJECT WHITE FEMALE MS

DATE 7-16-57

TOTAL INTENSIVE 245960

MEAN INTENSIVE 6675

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PLAN | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 524.96 | 300. | 0.121 | 19 | 1318.57 | 3168. | 1.269 |
| 2 | 635.60 | 6932. | 2.749 | 20 | 1335.63 | 10528. | 4.253 |
| 3 | 675.17 | 765. | 0.311 | 21 | 1353.06 | 3760. | 1.522 |
| 4 | 735.04 | 1792. | 0.724 | 22 | 1381.30 | 5436. | 2.403 |
| 5 | 805.03 | 4560. | 1.837 | 23 | 1391.76 | 6840. | 2.759 |
| 6 | 825.03 | 4712. | 1.904 | 24 | 1417.13 | 2560. | 1.037 |
| 7 | 891.66 | 3680. | 1.450 | 25 | 1435.62 | 3840. | 1.555 |
| 8 | 914.04 | 4088. | 1.638 | 26 | 1446.11 | 6624. | 2.682 |
| 9 | 922.83 | 7025. | 2.857 | 27 | 1479.87 | 5920. | 2.257 |
| 10 | 930.54 | 7680. | 3.110 | 28 | 1491.24 | 5920. | 2.257 |
| 11 | 1004.43 | 6755. | 2.749 | 29 | 1528.21 | 2914. | 1.174 |
| 12 | 1074.74 | 47520. | 19.240 | 30 | 1545.60 | 514. | 0.207 |
| 13 | 1121.91 | 26380. | 10.740 | 31 | 1560.73 | 84. | 0.026 |
| 14 | 1174.88 | 10138. | 4.150 | 32 | 1577.45 | 1280. | 0.518 |
| 15 | 1215.71 | 11775. | 4.768 | 33 | 1587.75 | 792. | 0.321 |
| 16 | 1234.21 | 10415. | 4.217 | 34 | 1597.61 | 864. | 0.350 |
| 17 | 1254.41 | 4154. | 1.684 | 35 | 1613.95 | 4200. | 1.631 |
| 18 | 1274.05 | 15744. | 7.554 | 36 | 1625.57 | 2400. | 0.998 |
| | | | | 37 | 1638.66 | 864. | 0.350 |

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SUBJECT: APT-11 MC JC

DATE: 7-1-68 TOTAL INTEGRATION: 1014.0

| PEAK | RETENTION TIME | WAVE AREA | WAVE AREA | RETENTION TIME | WAVE AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------|----------------|-----------|-----------------|
| 1 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.742 |
| 2 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 3.604 |
| 3 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.940 |
| 4 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.023 |
| 5 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.798 |
| 6 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.369 |
| 7 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.176 |
| 8 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 4.258 |
| 9 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.116 |
| 10 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.577 |
| 11 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.910 |
| 12 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 4.770 |
| 13 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.100 |
| 14 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.037 |
| 15 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.000 |
| 16 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.039 |
| 17 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.550 |
| 18 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.083 |
| 19 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.023 |
| 20 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.103 |
| 21 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.181 |
| 22 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.277 |
| 23 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.724 |

SUBJECT: APT-11 MC MK

DATE: 7-1-68 TOTAL INTEGRATION: 1014.0

| PEAK | RETENTION TIME | WAVE AREA | WAVE AREA | RETENTION TIME | WAVE AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------|----------------|-----------|-----------------|
| 1 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.742 |
| 2 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 3.604 |
| 3 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.940 |
| 4 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.023 |
| 5 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.798 |
| 6 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.369 |
| 7 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.176 |
| 8 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 4.258 |
| 9 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.116 |
| 10 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.577 |
| 11 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.910 |
| 12 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 4.770 |
| 13 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.100 |
| 14 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.037 |
| 15 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.000 |
| 16 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.039 |
| 17 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.550 |
| 18 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.083 |
| 19 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.023 |
| 20 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.103 |
| 21 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 2.181 |
| 22 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 1.277 |
| 23 | 12.54 | 12.54 | 12.54 | 12.54 | 12.54 | 0.724 |

| SUBJECT | | DATE | | TIME | | SN | |
|---------|----------------|--------|---------|------------|------------|------------|------------|
| TOTAL | | TUNING | | FREQ | | WAVE | |
| PEAK | RETENTION TIME | AREA | PERCENT | WAVELENGTH | WAVELENGTH | WAVELENGTH | WAVELENGTH |
| 1 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 2 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 3 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 4 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 5 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 6 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 7 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 8 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 9 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 10 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 11 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 12 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 13 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 14 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 15 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 17 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 18 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |
| 19 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 | 1.16 |

NOT REPRODUCIBLE

WC

DATE

TOTAL

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| PEAK | RETENTION TIME | AREA | PLA AREA | NORMALIZED AREA |
|------|----------------|------|----------|-----------------|
| 1 | 1276.72 | 44 | 8004 | 0.431 |
| 2 | 1314.89 | 43 | 1064 | 0.435 |
| 3 | 1330.16 | 24 | 3756 | 2.556 |
| 4 | 1347.01 | 25 | 672 | 0.528 |
| 5 | 1357.78 | 26 | 336 | 0.264 |
| 6 | 1361.78 | 27 | 512 | 0.406 |
| 7 | 1377.80 | 28 | 2752 | 2.161 |
| 8 | 1387.13 | 29 | 5700 | 4.512 |
| 9 | 1413.46 | 30 | 624 | 0.490 |
| 10 | 1447.86 | 31 | 2408 | 1.734 |
| 11 | 1459.57 | 32 | 2496 | 1.960 |
| 12 | 1477.02 | 33 | 2400 | 1.884 |
| 13 | 1488.14 | 34 | 7400 | 5.872 |
| 14 | 1524.92 | 35 | 1656 | 1.300 |
| 15 | 1543.70 | 36 | 1648 | 1.291 |
| 16 | 1575.57 | 37 | 4592 | 3.605 |
| 17 | 1587.81 | 38 | 1300 | 1.021 |
| 18 | 1593.64 | 39 | 1756 | 1.384 |
| 19 | 1617.56 | 40 | 756 | 0.594 |
| 20 | 1675.57 | 41 | 2800 | 2.196 |
| 21 | 1677.34 | 42 | 1456 | 1.143 |

EO

DATE

TOTAL

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| PEAK | RETENTION TIME | AREA | PLA AREA | NORMALIZED AREA |
|------|----------------|------|----------|-----------------|
| 17 | 1631.10 | 17 | 4896 | 3.878 |
| 18 | 1673.08 | 20 | 1024 | 1.108 |
| 19 | 1688.90 | 21 | 3400 | 2.618 |
| 20 | 1700.34 | 22 | 3400 | 2.618 |
| 21 | 1724.51 | 23 | 1100 | 1.210 |
| 22 | 1807.02 | 24 | 3577 | 2.877 |
| 23 | 1814.72 | 25 | 4200 | 3.283 |
| 24 | 1843.54 | 26 | 1900 | 1.474 |
| 25 | 1858.92 | 27 | 1176 | 1.270 |
| 26 | 1872.78 | 28 | 1344 | 1.052 |
| 27 | 1886.23 | 29 | 336 | 0.263 |
| 28 | 1902.80 | 30 | 2156 | 1.689 |
| 29 | 1914.98 | 31 | 8192 | 6.448 |
| 30 | 1947.61 | 32 | 224 | 0.242 |
| 31 | 1957.23 | 33 | 344 | 0.268 |
| 32 | 1975.87 | 34 | 352 | 0.260 |
| 33 | 1991.49 | 35 | 280 | 0.204 |
| 34 | 1991.49 | 36 | 1456 | 1.143 |
| 35 | 1991.49 | 37 | 1742 | 1.375 |

SUBJECT: 44-38861-56

DATE: 10-12-67

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TOTAL INTENSIVE

| PEAK | RETENTION TIME | AREA | AREA | RETENTION TIME | AREA | AREA |
|------|----------------|------|-------|----------------|---------|------|
| 1 | 315.50 | 1170 | 11130 | 16 | 1071.75 | 1780 |
| 2 | 501.80 | 602 | 6180 | 17 | 1151.05 | 1840 |
| 3 | 600.00 | 526 | 5400 | 18 | 1091.75 | 1840 |
| 4 | 625.68 | 412 | 4250 | 19 | 1155.66 | 1840 |
| 5 | 695.95 | 408 | 4250 | 20 | 1165.82 | 1840 |
| 6 | 765.34 | 762 | 7800 | 21 | 1151.45 | 1840 |
| 7 | 811.87 | 1100 | 1100 | 22 | 1210.35 | 1840 |
| 8 | 835.82 | 710 | 710 | 23 | 1223.34 | 1840 |
| 9 | 841.75 | 280 | 280 | 24 | 1244.42 | 1840 |
| 10 | 918.51 | 1200 | 1200 | | | |
| 11 | 945.35 | 1600 | 1600 | | | |
| 12 | 965.90 | 408 | 408 | | | |
| 13 | 975.12 | 720 | 720 | | | |
| 14 | 1013.42 | 332 | 332 | | | |
| 15 | 1021.92 | 974 | 974 | | | |
| 16 | 1031.75 | 2940 | 2940 | | | |
| 17 | 1091.75 | 260 | 260 | | | |
| 18 | 1125.84 | 5184 | 5184 | | | |
| 19 | 1155.66 | 558 | 558 | | | |
| 20 | 1165.82 | 2154 | 2154 | | | |
| 21 | 1151.45 | 400 | 400 | | | |
| 22 | 1210.35 | 400 | 400 | | | |
| 23 | 1223.34 | 400 | 400 | | | |
| 24 | 1244.42 | 400 | 400 | | | |

SUBJECT: 44-38861-56

DATE: 10-12-67

56

TOTAL INTENSIVE

| PEAK | RETENTION TIME | AREA | AREA | RETENTION TIME | AREA | AREA |
|------|----------------|------|-------|----------------|---------|------|
| 1 | 315.50 | 1170 | 11130 | 16 | 1071.75 | 1780 |
| 2 | 501.80 | 602 | 6180 | 17 | 1151.05 | 1840 |
| 3 | 600.00 | 526 | 5400 | 18 | 1091.75 | 1840 |
| 4 | 625.68 | 412 | 4250 | 19 | 1155.66 | 1840 |
| 5 | 695.95 | 408 | 4250 | 20 | 1165.82 | 1840 |
| 6 | 765.34 | 762 | 7800 | 21 | 1151.45 | 1840 |
| 7 | 811.87 | 1100 | 1100 | 22 | 1210.35 | 1840 |
| 8 | 835.82 | 710 | 710 | 23 | 1223.34 | 1840 |
| 9 | 841.75 | 280 | 280 | 24 | 1244.42 | 1840 |
| 10 | 918.51 | 1200 | 1200 | | | |
| 11 | 945.35 | 1600 | 1600 | | | |
| 12 | 965.90 | 408 | 408 | | | |
| 13 | 975.12 | 720 | 720 | | | |
| 14 | 1013.42 | 332 | 332 | | | |
| 15 | 1021.92 | 974 | 974 | | | |
| 16 | 1031.75 | 2940 | 2940 | | | |
| 17 | 1091.75 | 260 | 260 | | | |
| 18 | 1125.84 | 5184 | 5184 | | | |
| 19 | 1155.66 | 558 | 558 | | | |
| 20 | 1165.82 | 2154 | 2154 | | | |
| 21 | 1151.45 | 400 | 400 | | | |
| 22 | 1210.35 | 400 | 400 | | | |
| 23 | 1223.34 | 400 | 400 | | | |
| 24 | 1244.42 | 400 | 400 | | | |

SUBJECT WHITE FEMALE PP
DATE 10-3-57
TOTAL INTENSIVE 14950.

63

PEAK INTENSIVE 3650.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 51.50 | 1415. | 0.274 | 19 | 1195.20 | 11200. | 7.863 |
| 2 | 54.50 | 545. | 0.092 | 20 | 1244.17 | 11424. | 8.021 |
| 3 | 54.50 | 545. | 0.092 | 21 | 1255.57 | 12688. | 1.887 |
| 4 | 71.50 | 584. | 0.094 | 22 | 1298.08 | 13440. | 9.436 |
| 5 | 77.50 | 432. | 0.083 | 23 | 1339.32 | 4048. | 2.842 |
| 6 | 80.50 | 544. | 0.092 | 24 | 1353.11 | 7176. | 5.038 |
| 7 | 83.50 | 2304. | 1.018 | 25 | 1369.70 | 2976. | 2.069 |
| 8 | 87.50 | 1632. | 0.714 | 26 | 1401.86 | 12096. | 8.492 |
| 9 | 92.50 | 864. | 0.407 | 27 | 1438.24 | 4536. | 3.185 |
| 10 | 95.50 | 1728. | 1.213 | 28 | 1465.84 | 5632. | 3.954 |
| 11 | 97.50 | 1968. | 1.362 | 29 | 1478.82 | 792. | 0.556 |
| 12 | 98.50 | 4320. | 3.033 | 30 | 1503.04 | 4694. | 3.294 |
| 13 | 103.50 | 1008. | 0.708 | 31 | 1511.76 | 2634. | 1.848 |
| 14 | 105.50 | 480. | 0.370 | 32 | 1535.73 | 840. | 0.590 |
| 15 | 106.50 | 3824. | 2.123 | 33 | 1553.37 | 2250. | 1.580 |
| 16 | 109.50 | 1540. | 0.717 | 34 | 1568.25 | 850. | 0.597 |
| 17 | 114.50 | 6512. | 3.976 | 35 | 1584.44 | 240. | 0.169 |
| 18 | 116.50 | 9600. | 5.556 | 36 | 1602.26 | 2376. | 1.668 |
| | | | | 37 | 1619.13 | 1028. | 0.722 |

SUBJECT WHITE FEMALE FS
DATE 10-3-57
TOTAL INTENSIVE 57510.

64

PEAK INTENSIVE 15087.

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 51.50 | 476. | 0.074 | 20 | 1242.70 | 13312. | 2.322 |
| 2 | 50.50 | 344. | 0.060 | 21 | 1285.02 | 5700. | 1.005 |
| 3 | 67.50 | 324. | 0.057 | 22 | 1300.23 | 15720. | 4.268 |
| 4 | 71.50 | 1020. | 0.335 | 23 | 1313.43 | 6528. | 1.839 |
| 5 | 80.50 | 990. | 0.173 | 24 | 1333.13 | 3572. | 0.823 |
| 6 | 83.50 | 1440. | 0.251 | 25 | 1353.58 | 11440. | 1.995 |
| 7 | 85.50 | 712. | 0.124 | 26 | 1369.19 | 5888. | 1.027 |
| 8 | 89.50 | 2424. | 0.423 | 27 | 1385.87 | 9588. | 1.669 |
| 9 | 92.50 | 4280. | 0.714 | 28 | 1398.04 | 5824. | 1.016 |
| 10 | 95.50 | 4760. | 0.829 | 29 | 1411.48 | 26942. | 4.899 |
| 11 | 97.50 | 2012. | 0.355 | 30 | 1438.25 | 7616. | 1.328 |
| 12 | 100.50 | 2568. | 0.456 | 31 | 1453.54 | 5544. | 0.967 |
| 13 | 104.50 | 4920. | 0.857 | 32 | 1465.40 | 15776. | 2.752 |
| 14 | 105.50 | 14520. | 2.505 | 33 | 1479.79 | 4032. | 0.703 |
| 15 | 108.50 | 37224. | 6.495 | 34 | 1514.80 | 35228. | 6.155 |
| 16 | 114.50 | 2744. | 0.483 | 35 | 1545.58 | 4320. | 0.754 |
| 17 | 116.50 | 2376. | 0.419 | 36 | 1583.10 | 7680. | 1.340 |
| 18 | 119.50 | 4480. | 0.786 | 37 | 1605.07 | 5104. | 0.890 |
| 19 | 121.50 | 10216. | 1.784 | 38 | 1618.40 | 3472. | 0.606 |

I-32

65

JK

SUBJECT

DATE

TOTAL INTENSIVE

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

65%

TOTAL INTENSIVE

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 345.99 | 609. | 0.410 | 20 | 1274.15 | 12640. | 8.630 |
| 2 | 423.79 | 656. | 0.448 | 21 | 1275.78 | 8424. | 5.751 |
| 3 | 527.54 | 603. | 0.410 | 22 | 1498.69 | 12480. | 8.241 |
| 4 | 600.00 | 1240. | 0.847 | 23 | 1348.48 | 2856. | 1.950 |
| 5 | 629.37 | 1440. | 0.983 | 24 | 1363.77 | 2112. | 1.442 |
| 6 | 720.79 | 1064. | 0.724 | 25 | 1398.69 | 2880. | 1.966 |
| 7 | 755.41 | 800. | 0.545 | 26 | 1405.15 | 3420. | 2.335 |
| 8 | 800.00 | 680. | 0.467 | 27 | 1433.07 | 1360. | 0.929 |
| 9 | 822.22 | 4016. | 0.694 | 28 | 1449.11 | 1512. | 1.032 |
| 10 | 850.75 | 928. | 0.634 | 29 | 1461.59 | 1634. | 1.114 |
| 11 | 895.67 | 4895. | 3.343 | 30 | 1497.54 | 1474. | 1.006 |
| 12 | 913.55 | 560. | 1.746 | 31 | 1508.65 | 4940. | 3.373 |
| 13 | 961.26 | 4800. | 3.277 | 32 | 1537.46 | 1320. | 0.901 |
| 14 | 980.56 | 13056. | 8.914 | 33 | 1546.67 | 1470. | 1.004 |
| 15 | 1021.22 | 15680. | 10.705 | 34 | 1564.54 | 360. | 0.246 |
| 16 | 1077.55 | 4800. | 3.277 | 35 | 1583.16 | 132. | 0.090 |
| 17 | 1103.61 | 3480. | 2.376 | 36 | 1599.50 | 1604. | 1.136 |
| 18 | 1141.85 | 10080. | 6.862 | 37 | 1614.65 | 4104. | 2.802 |
| 19 | 1194.74 | 4792. | 3.665 | 38 | 1636.64 | 2520. | 1.721 |

66

CK

SUBJECT

DATE

TOTAL INTENSIVE

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

66%

TOTAL INTENSIVE

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

RETENTION TIME

PEAK AREA

NORMALIZED AREA

PEAK

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 362.65 | 1328. | 0.713 | 20 | 1246.03 | 5904. | 3.170 |
| 2 | 558.59 | 2352. | 1.264 | 21 | 1266.95 | 1968. | 1.057 |
| 3 | 627.56 | 1032. | 0.554 | 22 | 1297.00 | 10080. | 5.212 |
| 4 | 712.65 | 1512. | 0.812 | 23 | 1312.54 | 3520. | 1.890 |
| 5 | 774.53 | 1424. | 0.768 | 24 | 1332.51 | 3168. | 1.701 |
| 6 | 827.75 | 2176. | 1.168 | 25 | 1349.89 | 6160. | 3.307 |
| 7 | 867.97 | 3560. | 1.965 | 26 | 1364.38 | 2976. | 1.598 |
| 8 | 938.34 | 1056. | 0.567 | 27 | 1400.00 | 11040. | 5.927 |
| 9 | 985.93 | 4192. | 2.251 | 28 | 1444.51 | 4536. | 2.435 |
| 10 | 1013.10 | 1792. | 0.962 | 29 | 1455.21 | 4104. | 2.203 |
| 11 | 1065.14 | 2592. | 1.392 | 30 | 1477.63 | 3248. | 1.744 |
| 12 | 1083.96 | 3500. | 1.879 | 31 | 1461.93 | 3608. | 2.044 |
| 13 | 1098.43 | 1820. | 0.977 | 32 | 1498.49 | 13624. | 7.422 |
| 14 | 1124.63 | 6480. | 3.479 | 33 | 1506.80 | 8320. | 4.467 |
| 15 | 1133.62 | 2368. | 1.271 | 34 | 1521.03 | 2112. | 1.134 |
| 16 | 1170.72 | 4032. | 2.165 | 35 | 1546.84 | 21120. | 11.339 |
| 17 | 1194.82 | 8736. | 4.690 | 36 | 1598.27 | 9296. | 4.991 |
| 18 | 1214.91 | 1792. | 0.962 | 37 | 1610.56 | 1120. | 0.601 |
| 19 | 1230.31 | 2800. | 1.503 | 38 | 1615.67 | 2592. | 1.392 |
| | | | | 39 | 1634.86 | 6720. | 3.608 |

I-33

SUBJECT WHITE MALE FO

DATE 10-18-67

TOTAL INTENSIVE

PEAK INTENSIVE

1961

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 30.85 | 524 | 1.000 | 22 | 1229.78 | 1700 | 3.012 |
| 2 | 43.21 | 174 | 1.010 | 23 | 1242.27 | 1152 | 1.972 |
| 3 | 505.87 | 744 | 1.273 | 24 | 1268.29 | 476 | 0.815 |
| 4 | 603.00 | 400 | 0.687 | 25 | 1298.66 | 1872 | 3.204 |
| 5 | 674.22 | 420 | 0.711 | 26 | 1330.59 | 140 | 0.240 |
| 6 | 712.85 | 1524 | 1.754 | 27 | 1358.51 | 112 | 0.192 |
| 7 | 767.63 | 552 | 0.944 | 28 | 1349.43 | 612 | 1.048 |
| 8 | 811.39 | 1008 | 1.725 | 29 | 1365.94 | 468 | 0.801 |
| 9 | 855.89 | 552 | 0.944 | 30 | 1400.00 | 1456 | 2.592 |
| 10 | 901.14 | 480 | 0.826 | 31 | 1403.84 | 1872 | 3.204 |
| 11 | 955.12 | 720 | 1.231 | 32 | 1436.44 | 832 | 1.424 |
| 12 | 969.49 | 456 | 0.801 | 33 | 1449.23 | 792 | 1.356 |
| 13 | 995.27 | 448 | 0.784 | 34 | 1463.03 | 2080 | 3.550 |
| 14 | 1012.86 | 1148 | 2.013 | 35 | 1475.96 | 150 | 0.257 |
| 15 | 1022.52 | 510 | 0.877 | 36 | 1479.10 | 308 | 0.527 |
| 16 | 1064.78 | 2448 | 4.271 | 37 | 1500.09 | 1950 | 3.338 |
| 17 | 1091.55 | 2520 | 4.437 | 38 | 1508.31 | 2780 | 4.840 |
| 18 | 1120.82 | 3120 | 5.342 | 39 | 1538.08 | 1232 | 2.109 |
| 19 | 1145.34 | 3524 | 6.170 | 40 | 1548.44 | 1536 | 2.624 |
| 20 | 1169.13 | 1308 | 2.304 | 41 | 1592.50 | 2760 | 4.724 |
| 21 | 1182.62 | 2556 | 4.488 | 42 | 1617.72 | 1368 | 2.392 |

SUBJECT WHITE MALE BK

DATE 10-11-67

TOTAL INTENSIVE

PEAK INTENSIVE

1968

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 399.69 | 2900 | 0.769 | 19 | 1220.97 | 10472 | 3.040 |
| 2 | 500.00 | 2144 | 0.612 | 20 | 1234.33 | 22176 | 6.436 |
| 3 | 553.71 | 1880 | 0.521 | 21 | 1254.87 | 9856 | 2.861 |
| 4 | 734.95 | 2080 | 0.574 | 22 | 1260.59 | 36080 | 10.475 |
| 5 | 807.51 | 848 | 0.236 | 23 | 1305.23 | 9784 | 2.844 |
| 6 | 893.64 | 1248 | 0.346 | 24 | 1341.13 | 29440 | 8.547 |
| 7 | 971.73 | 1920 | 0.537 | 25 | 1358.47 | 9056 | 2.651 |
| 8 | 984.52 | 2368 | 0.657 | 26 | 1360.03 | 28800 | 8.361 |
| 9 | 1012.89 | 3360 | 0.935 | 27 | 1528.44 | 12096 | 3.524 |
| 10 | 1016.89 | 12000 | 3.484 | 28 | 1544.10 | 624 | 0.182 |
| 11 | 1043.83 | 10416 | 3.024 | 29 | 1548.92 | 56 | 0.016 |
| 12 | 1070.68 | 5632 | 1.568 | 30 | 1551.18 | 16 | 0.004 |
| 13 | 1091.55 | 17456 | 4.840 | 31 | 1571.77 | 36 | 0.010 |
| 14 | 1112.82 | 2336 | 0.648 | 32 | 1588.41 | 144 | 0.041 |
| 15 | 1135.15 | 2400 | 0.664 | 33 | 1571.94 | 416 | 0.120 |
| 16 | 116.14 | 11760 | 3.272 | 34 | 1543.02 | 6368 | 1.853 |
| 17 | 1185.04 | 21120 | 5.840 | 35 | 1607.74 | 10400 | 3.040 |
| 18 | 1204.37 | 11072 | 3.024 | 36 | 1630.77 | 4096 | 1.169 |

| SUBJECT | | DATE | | SH | | 69 | |
|-----------------|----------------|-----------|-----------------|--------|----------------|-----------|-----------------|
| TOTAL INTENSIVE | | 7-20-67 | | 195596 | | 3705 | |
| Mean Intensive | | 3705 | | | | | |
| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
| 1 | 127.46 | 1792. | 1.152 | 22 | 1257.92 | 4800. | 3.085 |
| 2 | 525.80 | 9216. | 1.067 | 23 | 1281.79 | 1732. | 0.792 |
| 3 | 630.04 | 1200. | 0.771 | 24 | 1291.06 | 960. | 0.617 |
| 4 | 653.84 | 2086. | 1.342 | 25 | 1308.50 | 4592. | 2.951 |
| 5 | 731.72 | 1344. | 0.864 | 26 | 1319.68 | 2976. | 1.913 |
| 6 | 747.32 | 1232. | 0.792 | 27 | 1344.82 | 800. | 0.514 |
| 7 | 811.87 | 2424. | 1.558 | 28 | 1360.04 | 1662. | 1.068 |
| 8 | 833.82 | 960. | 0.617 | 29 | 1378.75 | 640. | 0.411 |
| 9 | 853.74 | 1792. | 1.152 | 30 | 1411.15 | 3200. | 2.057 |
| 10 | 918.91 | 784. | 0.504 | 31 | 1419.90 | 2240. | 1.440 |
| 11 | 951.48 | 2256. | 1.450 | 32 | 1446.33 | 2580. | 1.658 |
| 12 | 985.01 | 10880. | 6.992 | 33 | 1462.92 | 2632. | 1.692 |
| 13 | 1004.53 | 640. | 0.411 | 34 | 1473.13 | 2632. | 1.692 |
| 14 | 1037.57 | 3960. | 2.545 | 35 | 1491.29 | 364. | 0.234 |
| 15 | 1069.31 | 4976. | 3.193 | 36 | 1511.98 | 2934. | 1.886 |
| 16 | 1088.02 | 7872. | 5.059 | 37 | 1520.85 | 3008. | 1.933 |
| 17 | 1120.99 | 5760. | 3.702 | 38 | 1550.37 | 1200. | 0.771 |
| 18 | 1158.03 | 2720. | 1.748 | 39 | 1559.97 | 504. | 0.324 |
| 19 | 1174.30 | 3952. | 2.540 | 40 | 1573.01 | 720. | 0.463 |
| 20 | 1205.21 | 6496. | 4.175 | 41 | 1589.95 | 120. | 0.077 |
| 21 | 1238.41 | 6912. | 4.542 | 42 | 1614.57 | 45544. | 28.628 |

SUBJECT AIR SAMPLE MACHINE SHOP (PHYSICS) 70
 DATE 10-24-67
 TOTAL INTENSITY 32730.0
 HEAD INTENSITY 9352.0

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 197.61 | 432. | 0.132 | 18 | 1230.96 | 20008. | 6.113 |
| 2 | 425.41 | 6720. | 2.053 | 19 | 1256.05 | 920. | 0.281 |
| 3 | 525.27 | 3850. | 1.173 | 20 | 1289.00 | 34450. | 10.537 |
| 4 | 728.40 | 1920. | 0.587 | 21 | 1300.00 | 5600. | 1.711 |
| 5 | 773.82 | 3520. | 1.075 | 22 | 1316.06 | 5760. | 1.760 |
| 6 | 810.95 | 980. | 0.293 | 23 | 1337.39 | 8928. | 2.728 |
| 7 | 831.34 | 1732. | 0.527 | 24 | 1351.54 | 1920. | 0.587 |
| 8 | 875.34 | 5080. | 1.552 | 25 | 1355.08 | 21504. | 6.570 |
| 9 | 927.50 | 6132. | 1.873 | 26 | 1421.57 | 2496. | 0.753 |
| 10 | 965.95 | 20195. | 6.173 | 27 | 1448.76 | 8600. | 2.648 |
| 11 | 1000.00 | 4800. | 1.465 | 28 | 1480.78 | 8352. | 2.552 |
| 12 | 1016.31 | 26780. | 8.243 | 29 | 1500.80 | 3120. | 0.953 |
| 13 | 1073.79 | 24304. | 7.425 | 30 | 1527.18 | 3404. | 1.040 |
| 14 | 1126.48 | 26880. | 8.212 | 31 | 1541.52 | 3640. | 1.113 |
| 15 | 1157.53 | 11560. | 3.574 | 32 | 1557.65 | 600. | 0.183 |
| 16 | 1183.38 | 39680. | 12.123 | 33 | 1590.82 | 4896. | 1.496 |
| 17 | 1201.55 | 6400. | 1.955 | 34 | 1608.02 | 960. | 0.293 |
| | | | | 35 | 1617.00 | 528. | 0.161 |

SUBJECT LABORATORY
DATE 12/10/72
TOTAL INVENTORY 100

1. The first group of people who are not allowed to enter the country are those who are not citizens of the United States.

[illegible]

| SUBJECT | LOCATION | DATE | TIME | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|---------|----------|------|------|----------------|-----------|-----------------|
| 1 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 2 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 3 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 4 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 5 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 6 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 7 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 8 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 9 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 10 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 11 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 12 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 13 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 14 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 15 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 16 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 17 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 18 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| 19 | 50.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |

SUBJECT TROPICAL PLANT 2ND EXPERIMENT
 DATE 12-15-64
 TOTAL INTENSITY 7774

| PEAK | RETENTION TIME | PEAK AREA | CORRECTED AREA | PEAK | RETENTION TIME | PEAK AREA | CORRECTED AREA |
|------|----------------|-----------|----------------|------|----------------|-----------|----------------|
| 1 | 40.17 | 249 | 0.09 | 24 | 1318.01 | 4280 | 5.138 |
| 2 | 51.26 | 142 | 0.16 | 25 | 1379.10 | 3714 | 4.254 |
| 3 | 53.37 | 84 | 0.09 | 26 | 1453.58 | 4086 | 4.871 |
| 4 | 71.13 | 125 | 1.514 | 27 | 1529.27 | 1656 | 1.883 |
| 5 | 74.34 | 432 | 2.893 | 28 | 1551.80 | 2304 | 2.627 |
| 6 | 81.11 | 542 | 2.621 | 29 | 1574.91 | 1252 | 1.455 |
| 7 | 81.53 | 74 | 0.802 | 30 | 1616.08 | 140 | 0.146 |
| 8 | 85.13 | 176 | 0.123 | 31 | 1626.01 | 3712 | 4.232 |
| 9 | 85.44 | 442 | 3.232 | 32 | 1635.03 | 2304 | 2.627 |
| 10 | 91.22 | 252 | 1.648 | 33 | 1652.97 | 1640 | 1.883 |
| 11 | 93.17 | 418 | 0.474 | 34 | 1672.35 | 3444 | 4.097 |
| 12 | 95.44 | 1342 | 1.494 | 35 | 1673.16 | 1680 | 1.883 |
| 13 | 95.58 | 6132 | 4.121 | 36 | 1688.12 | 210 | 0.246 |
| 14 | 102.65 | 1476 | 1.781 | 37 | 1692.54 | 336 | 0.351 |
| 15 | 125.21 | 140 | 0.735 | 38 | 1712.78 | 1420 | 1.619 |
| 16 | 135.17 | 1364 | 1.532 | 39 | 1724.72 | 4832 | 4.597 |
| 17 | 139.71 | 542 | 0.894 | 40 | 1737.81 | 1584 | 1.883 |
| 18 | 141.91 | 4112 | 1.144 | 41 | 1751.37 | 4800 | 4.472 |
| 19 | 147.29 | 2476 | 0.129 | 42 | 1765.15 | 560 | 0.526 |
| 20 | 173.33 | 3824 | 6.658 | 43 | 1778.56 | 1144 | 1.304 |
| 21 | 187.32 | 1216 | 1.385 | 44 | 1790.52 | 920 | 1.057 |
| 22 | 193.40 | 2736 | 3.119 | 45 | 1794.12 | 344 | 0.359 |
| 23 | 1277.59 | 2448 | 2.731 | 46 | 1825.57 | 2480 | 2.867 |

SUBJECT TROPICAL PLANT 2ND EXPERIMENT
 DATE 12-16-64
 TOTAL INTENSITY 74877

| PEAK | RETENTION TIME | PEAK AREA | CORRECTED AREA | PEAK | RETENTION TIME | PEAK AREA | CORRECTED AREA |
|------|----------------|-----------|----------------|------|----------------|-----------|----------------|
| 1 | 45.47 | 288 | 0.165 | 14 | 1522.25 | 4300 | 5.356 |
| 2 | 58.65 | 144 | 0.194 | 15 | 1532.15 | 1724 | 1.808 |
| 3 | 71.52 | 1552 | 1.452 | 16 | 1574.54 | 920 | 1.046 |
| 4 | 77.98 | 900 | 1.45 | 17 | 1613.42 | 3600 | 4.274 |
| 5 | 81.47 | 1048 | 1.400 | 18 | 1629.25 | 1600 | 1.817 |
| 6 | 82.54 | 482 | 1.344 | 19 | 1650.49 | 324 | 0.513 |
| 7 | 97.88 | 1700 | 1.657 | 20 | 1677.73 | 1120 | 1.257 |
| 8 | 97.88 | 1008 | 1.344 | 21 | 1678.13 | 740 | 0.844 |
| 9 | 97.88 | 1284 | 4.452 | 22 | 1678.58 | 880 | 1.044 |
| 10 | 100.43 | 282 | 1.14 | 23 | 1678.58 | 1680 | 1.883 |
| 11 | 101.34 | 1400 | 1.623 | 24 | 1678.58 | 344 | 0.352 |
| 12 | 102.07 | 744 | 1.32 | 25 | 1678.58 | 1120 | 1.257 |
| 13 | 103.75 | 1024 | 1.392 | 26 | 1678.58 | 1600 | 1.883 |
| 14 | 105.74 | 1064 | 2.428 | 27 | 1678.58 | 1600 | 1.883 |
| 15 | 110.84 | 976 | 2.428 | 28 | 1678.58 | 1600 | 1.883 |
| 16 | 112.00 | 1504 | 7.372 | 29 | 1678.58 | 2072 | 2.674 |
| 17 | 121.76 | 4264 | 6.64 | 30 | 1678.58 | 1600 | 1.883 |
| 18 | 123.41 | 1112 | 2.601 | 31 | 1678.58 | 1600 | 1.883 |

NOT REPRODUCIBLE

K01

SUBJECT KITCHEN 0000001

DATE 10-25-67

TOTAL INTENSITY 103000

WAVELENGTHS 40000

| PEAK | RETENTION TIME | PEAK AREA | UNNORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | UNNORMALIZED AREA |
|------|----------------|-----------|-------------------|------|----------------|-----------|-------------------|
| 1 | 555.45 | 1000 | 0.010 | 20 | 1219.67 | 62800 | 3.751 |
| 2 | 555.45 | 3000 | 0.030 | 21 | 1241.19 | 61400 | 3.450 |
| 3 | 555.45 | 1100 | 0.011 | 22 | 1253.92 | 28100 | 1.535 |
| 4 | 715.14 | 1100 | 0.011 | 23 | 1276.00 | 25600 | 1.376 |
| 5 | 751.60 | 6400 | 0.064 | 24 | 1291.42 | 26400 | 1.434 |
| 6 | 817.73 | 4000 | 0.040 | 25 | 1306.82 | 72000 | 3.948 |
| 7 | 855.78 | 21700 | 0.217 | 26 | 1353.11 | 29120 | 1.588 |
| 8 | 900.93 | 4000 | 0.040 | 27 | 1370.14 | 44800 | 2.424 |
| 9 | 924.59 | 17200 | 0.172 | 28 | 1410.95 | 112300 | 6.124 |
| 10 | 945.63 | 17300 | 0.173 | 29 | 1441.70 | 144000 | 7.785 |
| 11 | 957.64 | 25700 | 0.257 | 30 | 1465.53 | 27200 | 1.463 |
| 12 | 1000.91 | 40000 | 0.400 | 31 | 1510.19 | 67300 | 3.603 |
| 13 | 1055.24 | 18000 | 0.180 | 32 | 1524.62 | 64400 | 3.521 |
| 14 | 1092.06 | 20800 | 0.208 | 33 | 1538.10 | 39200 | 2.160 |
| 15 | 1102.20 | 79300 | 0.793 | 34 | 1546.15 | 12700 | 0.676 |
| 16 | 1121.91 | 207300 | 2.073 | 35 | 1566.24 | 21000 | 1.118 |
| 17 | 1154.91 | 113280 | 1.133 | 36 | 1579.62 | 36000 | 2.000 |
| 18 | 1184.23 | 105000 | 1.050 | 37 | 1599.39 | 158000 | 8.554 |
| 19 | 1205.28 | 115200 | 1.152 | 38 | 1609.56 | 238000 | 12.608 |

K02

SUBJECT KITCHEN 0000002

DATE 10-25-67

TOTAL INTENSITY 424242

WAVELENGTHS 114000

| PEAK | RETENTION TIME | PEAK AREA | UNNORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | UNNORMALIZED AREA |
|------|----------------|-----------|-------------------|------|----------------|-----------|-------------------|
| 1 | 629.40 | 32800 | 0.328 | 19 | 1241.44 | 211200 | 4.978 |
| 2 | 555.20 | 28400 | 0.284 | 20 | 1261.57 | 59200 | 1.403 |
| 3 | 752.71 | 79800 | 0.798 | 21 | 1281.05 | 76800 | 1.810 |
| 4 | 815.62 | 79200 | 0.792 | 22 | 1287.89 | 67400 | 1.569 |
| 5 | 934.35 | 52800 | 1.245 | 23 | 1314.17 | 135200 | 3.187 |
| 6 | 969.83 | 75300 | 1.775 | 24 | 1336.54 | 33600 | 0.794 |
| 7 | 985.55 | 45440 | 1.071 | 25 | 1354.26 | 18000 | 0.424 |
| 8 | 1013.45 | 147200 | 3.470 | 26 | 1372.07 | 82000 | 0.195 |
| 9 | 1024.62 | 128000 | 3.017 | 27 | 1383.59 | 100000 | 0.024 |
| 10 | 1042.68 | 169600 | 3.981 | 28 | 1409.20 | 256000 | 6.036 |
| 11 | 1057.54 | 232700 | 5.491 | 29 | 1440.04 | 82000 | 1.935 |
| 12 | 1061.25 | 576000 | 13.577 | 30 | 1463.05 | 83200 | 1.961 |
| 13 | 1109.27 | 158720 | 3.741 | 31 | 1506.32 | 341700 | 8.056 |
| 14 | 1121.91 | 355400 | 8.444 | 32 | 1535.44 | 112400 | 0.284 |
| 15 | 1173.11 | 424400 | 9.957 | 33 | 1543.55 | 48000 | 0.114 |
| 16 | 1185.73 | 230400 | 5.431 | 34 | 1559.44 | 42000 | 0.099 |
| 17 | 1212.68 | 103680 | 2.444 | 35 | 1593.46 | 50000 | 0.114 |
| 18 | 1243.43 | 110400 | 2.502 | 36 | 1604.82 | 87000 | 0.205 |
| | | | | 37 | 1625.57 | 192000 | 0.045 |

503

SUBJECT KITCHEN DOORS

DATE 10-27-50
TOTAL INTENSIVE 207.5

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 42.42 | 1792 | 6.052 | 22 | 1195.09 | 17192 | 6.052 |
| 2 | 50.00 | 1240 | 6.057 | 23 | 1224.19 | 17280 | 6.057 |
| 3 | 59.88 | 4228 | 6.013 | 24 | 1248.37 | 10240 | 6.013 |
| 4 | 73.04 | 3712 | 6.076 | 25 | 1273.74 | 5880 | 6.076 |
| 5 | 78.99 | 3936 | 6.638 | 26 | 1302.12 | 24480 | 6.638 |
| 6 | 80.03 | 3742 | 6.069 | 27 | 1313.47 | 5920 | 6.069 |
| 7 | 85.03 | 4672 | 6.439 | 28 | 1332.42 | 6412 | 6.439 |
| 8 | 92.45 | 4442 | 6.471 | 29 | 1357.84 | 9412 | 6.471 |
| 9 | 99.16 | 5472 | 6.589 | 30 | 1396.12 | 15048 | 6.589 |
| 10 | 114.4 | 5702 | 6.090 | 31 | 1427.87 | 5440 | 6.090 |
| 11 | 119.35 | 6112 | 6.686 | 32 | 1448.83 | 4788 | 6.686 |
| 12 | 124.35 | 6112 | 6.930 | 33 | 1472.93 | 8320 | 6.930 |
| 13 | 129.54 | 6602 | 6.084 | 34 | 1509.26 | 176 | 6.084 |
| 14 | 133.28 | 6742 | 6.719 | 35 | 1529.05 | 4872 | 6.719 |
| 15 | 135.82 | 6742 | 6.155 | 36 | 1541.88 | 440 | 6.155 |
| 16 | 139.97 | 6592 | 6.148 | 37 | 1547.97 | 560 | 6.148 |
| 17 | 1408.03 | 1542 | 6.169 | 38 | 1557.93 | 480 | 6.169 |
| 18 | 1445.65 | 1372 | 6.800 | 39 | 1576.26 | 2708 | 6.800 |
| 19 | 1461.91 | 9502 | 1.056 | 40 | 1577.43 | 2972 | 1.056 |
| 20 | 1495.81 | 9502 | 0.457 | 41 | 1577.43 | 1490 | 0.457 |
| 21 | 1495.81 | 13052 | 1.739 | 42 | 1625.57 | 4728 | 1.739 |

504

SUBJECT KITCHEN DOORS

DATE 10-27-50
TOTAL INTENSIVE 207.5

| PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA | PEAK | RETENTION TIME | PEAK AREA | NORMALIZED AREA |
|------|----------------|-----------|-----------------|------|----------------|-----------|-----------------|
| 1 | 313.94 | 342 | 6.070 | 16 | 1195.09 | 6340 | 6.070 |
| 2 | 52.41 | 564 | 6.197 | 17 | 1195.09 | 1928 | 6.197 |
| 3 | 60.00 | 422 | 6.221 | 18 | 1195.78 | 13120 | 6.221 |
| 4 | 65.14 | 182 | 6.414 | 19 | 1213.88 | 7504 | 6.414 |
| 5 | 73.42 | 3842 | 6.632 | 20 | 1235.40 | 14400 | 6.632 |
| 6 | 81.42 | 4372 | 6.274 | 21 | 1260.04 | 19320 | 6.274 |
| 7 | 83.42 | 3362 | 6.834 | 22 | 1271.43 | 2592 | 6.834 |
| 8 | 90.03 | 4622 | 1.812 | 23 | 1312.75 | 5632 | 1.812 |
| 9 | 92.45 | 4622 | 1.842 | 24 | 1330.14 | 5632 | 1.842 |
| 10 | 95.815 | 6122 | 1.945 | 25 | 1348.02 | 5988 | 1.945 |
| 11 | 97.05 | 7272 | 7.412 | 26 | 1357.09 | 23040 | 7.412 |
| 12 | 98.75 | 6432 | 5.712 | 27 | 1367.72 | 5712 | 5.712 |
| 13 | 101.44 | 13122 | 6.940 | 28 | 1377.78 | 2880 | 6.940 |
| 14 | 102.42 | 9982 | 1.861 | 29 | 1387.78 | 5040 | 1.861 |
| 15 | 104.52 | 5372 | 4.410 | 30 | 1443.77 | 13720 | 4.410 |
| 16 | 104.52 | 14072 | 5.770 | 31 | 1466.41 | 5520 | 5.770 |
| 17 | 107.41 | 17402 | 1.132 | 32 | 1545.72 | 3520 | 1.132 |
| | | 11132 | 6.516 | 33 | 1577.43 | 7808 | 6.516 |
| | | | | 34 | 1577.43 | 18588 | |

NOT REPRODUCIBLE

| SUBJECT | DATE | TIME | AREA | PERCENT | ADJUSTED AREA |
|---------|----------|-------|--------|---------|---------------|
| 1 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 2 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 3 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 4 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 5 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 6 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 7 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 8 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 9 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 10 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 11 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 12 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 13 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 14 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 15 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 16 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |
| 17 | 10/10/57 | 10:00 | 100.00 | 100.00 | 100.00 |

SUMMARY OF PATTERNS BY GROUPS

| Consecutive
Pattern No. | H U M A N S | | | | |
|----------------------------|-------------|---------|--------|-------|---------|
| | Caucasian | | Indian | Other | |
| | Males | Females | Males | Males | Females |
| 1 | | X | | | |
| 2 | | X | | | |
| 3 | | X | | | |
| 4 | | | X | | |
| 5 | | | X | | |
| 6 | | | X | | |
| 7 | | | X | | |
| 8 | | | X | | |
| 9 | | | X | | |
| 10 | | | X | | |
| 11 | | | X | | |
| 12 | | | X | | |
| 13 | X | | | | |
| 14 | X | | | | |
| 15 | X | | | | |
| 16 | X | | | | |
| 17 | | X | | | |
| 18 | X | | | | |
| 19 | X | | | | |
| 20 | X | | | | |
| 21 | X | | | | |
| 22 | X | | | | |
| 23 | X | | | | |
| 24 | | X | | | |
| 25 | | | | X | |
| 26 | X | | | | |
| 27 | | X | | | |
| 28 | | X | | | |
| 29 | | X | | | |
| 30 | | X | | | |
| 31 | | | | | X |
| 32 | | X | | | |
| 33 | | | | X | |
| 34 | | | | | X |
| 35 | | | | | X |
| 36 | X | | | | |
| 37 | X | | | | |
| 38 | X | | | | |
| 39 | X | | | | |
| 40 | X | | | | |
| 41 | X | | | | |
| 42 | X | | | | |
| 43 | X | | | | |
| 44 | X | | | | |
| 45 | | | X | | |
| 46 | | | X | | |
| 47 | | | X | | |
| 48 | X | | | | |
| 49 | | X | | | |
| 50 | | X | | | |
| 51 | | X | | | |
| 52 NOT USED | | | | | |
| 53 NOT USED | | | | | |
| 54 NOT USED | | | | | |
| 55 | | X | | | |
| 56 | | X | | | |
| 57 | | X | | | |
| 58 NOT USED | | | | | |
| 59 | X | | | | |
| 60 | X | | | | |
| 61 | | X | | | |
| 62 | | X | | | |
| 63 | | X | | | |
| 64 | | X | | | |
| 65 | X | | | | |
| 66 | | X | | | |
| 67 | | X | | | |
| 68 | X | | | | |
| 69 | | X | | | |
| Total | 25 | 23 | 12 | 2 | 3 |
| Environments | 10 | | | | |

APPENDIX II

Gas Chromatographic Positions of Peaks Obtained in Analysis of Vapors from the Same Caucasian Female

Analysis: Isothermal, 200-ft support-coated
open tubular column with Carbowax 20M.
Data are listed on Kovats Retention
Index Scale.

NOTES

- (1) For those ranges of the chromatograms which were outside of the calibrated range, the peak positions are given in minutes of retention time instead of Kovats Indexes.
- (2) Values below K.I. = 700 correspond to the very initial parts of the gas chromatograms and are much less exact (of order of ± 30 units at extreme early range) than the values in the higher K.I. ranges.
- (3) The last column represents difference in Kovats Indexes between peaks and shoulders observed in Run B and the closest matching peak in Run A. A plus (+) mark indicates that peaks in pattern B did not seem to have a reasonable counterpart in the pattern A within the limits of experimental error. A dash (-) means that a peak was observed in Run A but apparently had no satisfactory match in Run B.

Difference In
Kovats Indexes
Between Runs A & B
(See Note 3.)
Appendix Table Page 1

| Analysis Temperature, °C | | | | | | | |
|--------------------------|------|--------------|------|--------------|------|-----------------|-------------|
| 90° | | 120° | | 150° (A) | | 150° (B) Repeat | |
| Kovats Index | Min. | Kovats Index | Min. | Kovats Index | Min. | Kovats Index | Min. |
| | | | | 3.84 | | 3.84 | (0.00 min.) |
| | 4.13 | | | | | 4.04 | |
| | 4.26 | | | | | | |
| 435 | | | | | | | |
| 690 | | | | 692 | | | - |
| 648 | | | | | | | |
| 675 | | | | 697 | | | - |
| 715 | | | | | | | |
| | | 752 | | | | 730 | + (33?) |
| 758 | | | | | | | |
| 777 | | | | | | | |
| 798 | | | | | | | |
| 809 | | 803 | | 823 | | | |
| 837 | | | | | | | |
| | | 842 | | | | 844 | 4 |
| | | | | 848 | | | |
| 853 | | | | | | 867 | 14 |
| | | 870 | | | | | |
| 872 | | | | 881 | | | |
| 888 | | | | | | | |
| | | 891 | | | | | |
| | | 902 | | | | | |
| 903 | | | | 909 | | | |
| 910 | | | | | | | |
| | | 916 | | | | 916 | 7 |
| 927 | | 938 | | | | | |
| | | | | 942 | | | |
| 945 | | | | | | | |
| | | 954 | | | | 955 | 13 |
| 963 | | | | | | | |
| | | 967 | | | | | |
| 977 | | | | 969 | | | - |
| | | 981 | | | | | |
| | | 995 | | | | | |
| | | | | 999 | | | - |
| 1005 | | 1005 | | | | | |
| 1038 | | | | | | 1010 | 6 |
| | | 1013 | | | | | |
| | | | | 1016 | | | |
| 1017 | | | | | | | |
| 1024 | | 1024 | | | | | |
| 1031 | | | | 1031 | | | - |
| 1035 | | | | | | | |
| 1037 | | | | | | | |
| | | 1038 | | | | | |
| 1047 | | | | | | | |
| 1057 | | | | | | | |
| | | 1059 | | | | | |
| | | | | 1066 | | | |
| | | | | | | 1070 | 4 |
| | | 1071 | | | | | |
| 1074 | | | | | | | |
| 1076 | | | | | | | |
| | | 1077 | | | | | |
| 1082 | | | | | | | |
| | | 1088 | | | | | |
| 1089 | | | | | | | |
| 1093 | | | | | | | |
| 1096 | | | | | | | |
| 1101 | | | | | | | |
| | | 1108 | | | | | |
| 1109 | | | | | | | |
| | | 1116 | | | | | |
| 1122 | | | | | | | |
| 1127 | | | | 1130 | | | |

Difference In
Kovats Indexes
Between Runs A & B
(See Note 1.)

Appendix Title Page

| Analysis Temperature, °C | | | | | | | |
|--------------------------|-------|-----------------|------|-----------------|------|-----------------|------|
| 90° | | 120° | | 150° (A) | | 150° (B) Repeat | |
| Kovats
Index | Min. | Kovats
Index | Min. | Kovats
Index | Min. | Kovats
Index | Min. |
| 1131 | | 1131 | | | | 1131 | |
| 1149 | | | | | | | |
| 1143 | | | | 1147 | | | |
| 1151 | | 1147 | | | | | |
| | | 1153 | | | | | |
| 1166 | | | | | | 1160 | |
| 1175 | | 1150 | | | | | |
| | 11.95 | 1184 | | 1188 | | 1184 | |
| | | 1199 | | | | 1207 | |
| | | 1209 | | | | | |
| | | 1215 | | | | | |
| | | 1225 | | 1224 | | | |
| | | 1236 | | | | 1226 | |
| | | 1247 | | 1247 | | 1246 | |
| | | 1259 | | | | | |
| | | | | 1266 | | 1262 | |
| | | 1274 | | | | 1276 | |
| | | 1280 | | | | | |
| | | 1290 | | | | 1291 | |
| | | | | 1293 | | | |
| | | 1300 | | | | | |
| | | 1308 | | 1311 | | | |
| | | 1313 | | | | 1316 | |
| | | | | 1320 | | | |
| | | 1321 | | | | | |
| | | 1326 | | 1332 | | | |
| | | 1335 | | | | 1336 | |
| | | 1340 | | | | | |
| | | 1348 | | 1350 | | 1350 | |
| | | 1352 | | | | | |
| | | 1353 | | | | | |
| | | 1360 | | | | | |
| | | | | 1364 | | 1365 | |
| | | 1368 | | | | | |
| | | 1379 | | 1374 | | | |
| | | 1386 | | | | 1380 | |
| | | | | 1388 | | | |
| | | | | | | 1389 | |
| | | 1390 | | | | | |
| | | 1393 | | | | | |
| | | 1395 | | | | | |
| | | 1398 | | | | 1399 | |
| | | | | 1400 | | | |
| | | 1405 | | | | | |
| | | | | 1414 | | 1412 | |
| | | 1415 | | | | | |
| | | | | 1421 | | 1418 | |
| | | 1432 | | | | | |
| | | | | 1428 | | 1428 | |
| | | | | 1439 | | 1435 | |
| | | 1442 | | | | | |
| | | | | 1452 | | 1451 | |
| | | 1453 | | | | | |
| | | | | | | 1457 | |

Difference In
Kovats Indexes
Between Runs A & B
(See Note 3,
Appendix Title Page)

| 90° | | 120° | | 150° (A) | | 150° (B) Repeat | |
|-----------------|------|-----------------|------|-----------------|------|-----------------|------|
| Kovats
Index | Min. | Kovats
Index | Min. | Kovats
Index | Min. | Kovats
Index | Min. |
| 1536 | | 1468 | | 1468 | | - | |
| | | | | 1470 | | 1 | |
| | | | | 1476 | | | |
| | | 1479 | | | | 1476 | |
| | | | | 1480 | | 1 | |
| | | | | 1481 | | | |
| | | | | 1488 | | 1 | |
| | | | | 1495 | | 0 | |
| | | | | 1500 | | | |
| | | | | | | 1502 | |
| | | | | | | 1508 | |
| | | | | 1510 | | 2 | |
| | | | | | | 1516 | |
| | | | | 1517 | | 1 | |
| | | | | 1524 | | - | |
| | | | | | | 1524 | |
| | | | | | | 1532 | |
| | | | | | | + | |
| | | | | | | 1544 | |
| | | | | | | 1551 | |
| | | | | | | 1558 | |
| | | | | 1560 | | 2 | |
| | | | | | | 1565 | |
| | | | | | | 1569 | |
| | | 1572 | | 1570 | | 1 | |
| | | | | | | 1574 | |
| | | | | 1577 | | + | |
| | | | | | | 1577 | |
| | | | | | | 1584 | |
| | | | | | | 1589 | |
| | | | | 1590 | | 1 | |
| | | | | | | 1595 | |
| | | | | 1597 | | 2 | |
| | | | | | | 1605 | |
| | | 1611 | | 1611 | | 3 | |
| | | | | | | 1618 | |
| | | | | 1620 | | 2 | |
| | | | | | | - (?) | |
| | | | | | | 1630 | |
| | | | | | | 1635 | |
| | | | | | | 1 | |
| | | | | 1634 | | | |
| | | | | | | 1642 | |
| | | | | 1645 | | 3 | |
| | | | | | | 1652 | |
| | | | | 1654 | | 2 | |
| | | | | | | 1658 | |
| | | | | 1660 | | 2 | |
| | | | | | | 1678 | |
| | | | | 1681 | | 3 | |
| | | | | 1687 | | | |
| | | | | | | 1688 | |
| | | | | 1690 | | 1 | |
| | | | | | | 1699 | |
| | | | | 1701 | | 2 | |
| | | | | 1702 | | - | |
| | | | | | | 1713 | |
| | | | | 1719 | | + | |
| | | | | | | 1721 | |
| | | | | | | 1733 | |
| | | | | 1736 | | 2 | |
| | | | | | | 1744 | |
| | | | | 1747 | | 3 | |
| | | | | 1754 | | | |
| | | | | | | 1759 | |
| | | | | | | 1762 | |
| | | | | 1765 | | 5 | |
| | | | | 1767 | | 3 | |
| | | | | 1769 | | - | |
| | | | | | | - | |
| | | | | | | 1780 | |
| | | | | | | 1781 | |
| | | | | 1782 | | 1 | |
| | | | | 1783 | | - | |
| | | | | 1784 | | - | |
| | | | | 1786 | | | |
| | | | | 1810 | | 1790 | |
| | | | | | | 4 | |
| | | | | | | 1811 | |
| | | | | | | 1813 | |
| | | | | | | 1830 | |

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| IIT Research Institute
Technology Center
Chicago, Illinois 60616 | | Unclassified | |
| 3. REPORT TYPE | | 26. GROUP | |
| DETECTION AND IDENTIFICATION OF CHEMICAL SIGNATURES | | | |
| 4. DESCRIPTIVE NOTES (Type of report and inclusive dates) | | | |
| 5. AUTHOR(S) (Print name, Eddies initial, Last name) | | | |
| A. Dravnicki and B. K. Krotoszynski | | | |
| 6. REPORT DATE | | 76. TOTAL NO. OF PAGES | 76. NO. OF REFS |
| May 19, 1969 | | 171 | |
| 80. CONTRACT OR GRANT NO. | | 90. ORIGINATOR'S REPORT NUMBER(S) | |
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| <p>In the work on the detection and identification of human chemical signatures, experimental and computerized statistical techniques were first developed, tested, and standardized. The experimental techniques solved the problems of sample collection, recovery, injection into the gas chromatograph, calibration and characterization of signature components. The sample collections were based on the principle of equilibration and were performed by means of Apiezon L-coated Teflon powder in the form of a fluidized bed. The collected samples were transferred into a special injector needle by means of an IITRI-designed transfer system. The injection of samples into the gas chromatograph was performed in a timed, automatic and reproducible manner with a specially constructed injection system. The calibration method permitted correlation of the concentrations of known signature components in air with their respective peak areas. The sensitivity of this novel process is of the order of one part per billion (by volume) of an organic component in air.</p> | | | |

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Chemical Signatures - Identification
Gas Chromatography | | | | | | |

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